SPECIAL ISSUE—RHEOLOGICAL TECHNIQUES

LABORATORY PRACTICE

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VOL. 10 No. 11 Published Monthly NOVEMBER 1961

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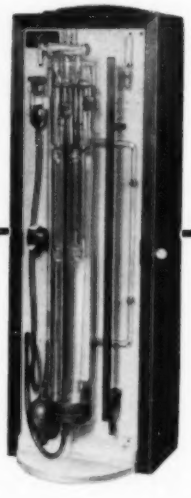

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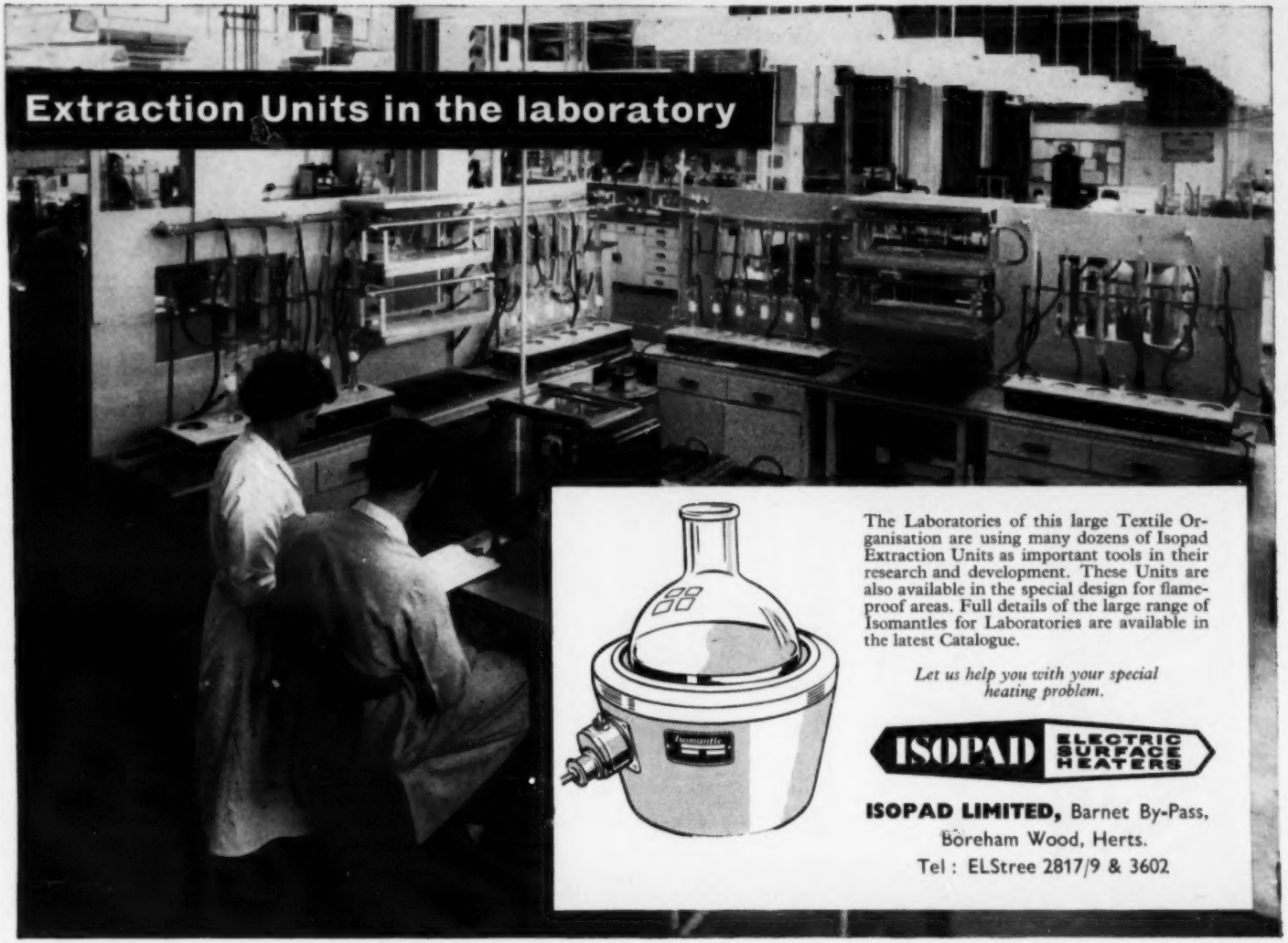

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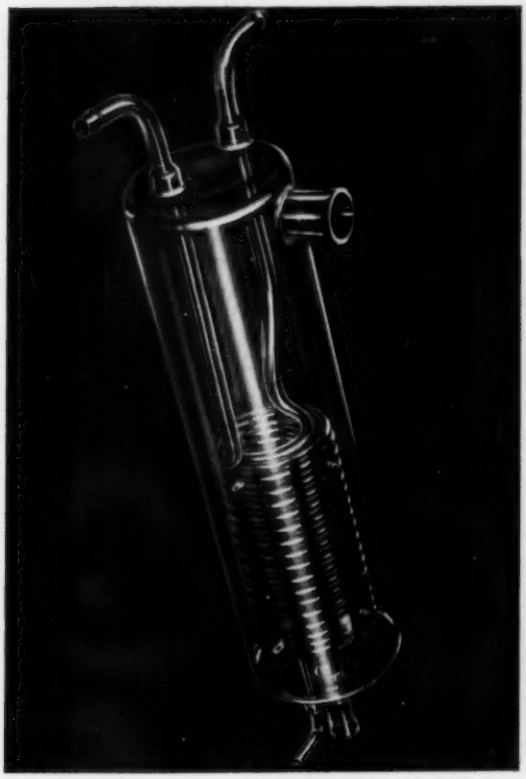

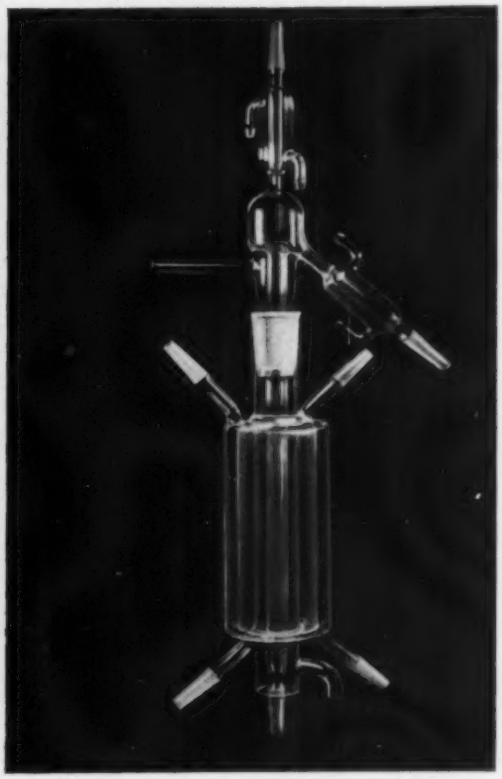

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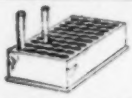

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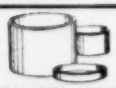

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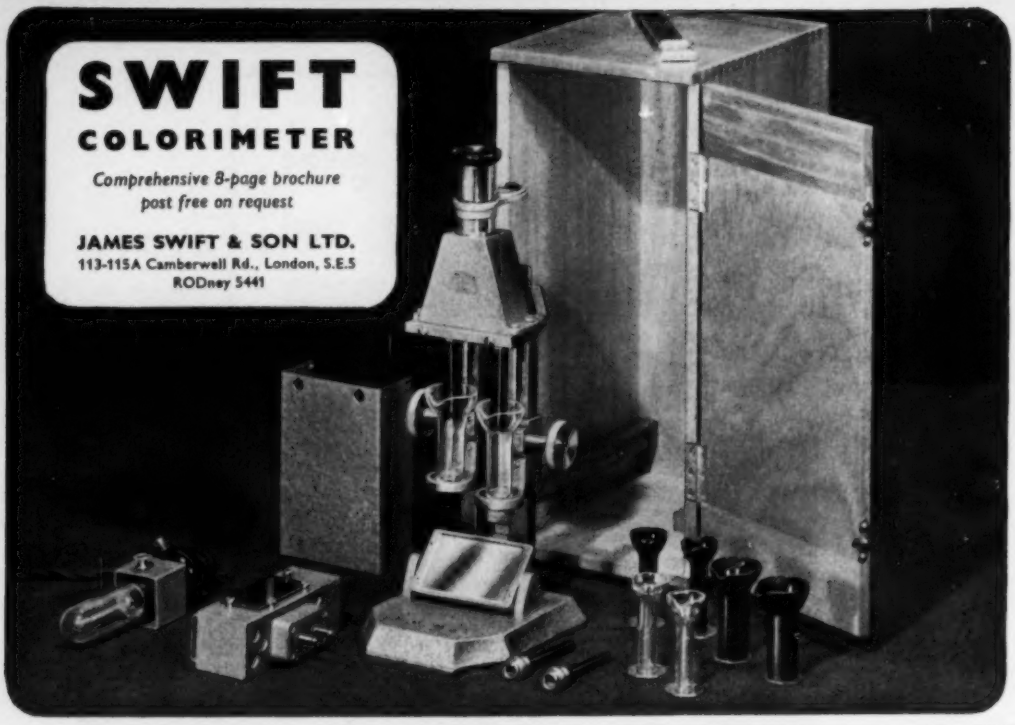

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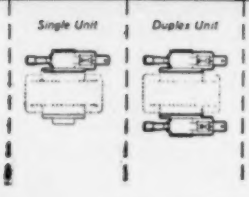

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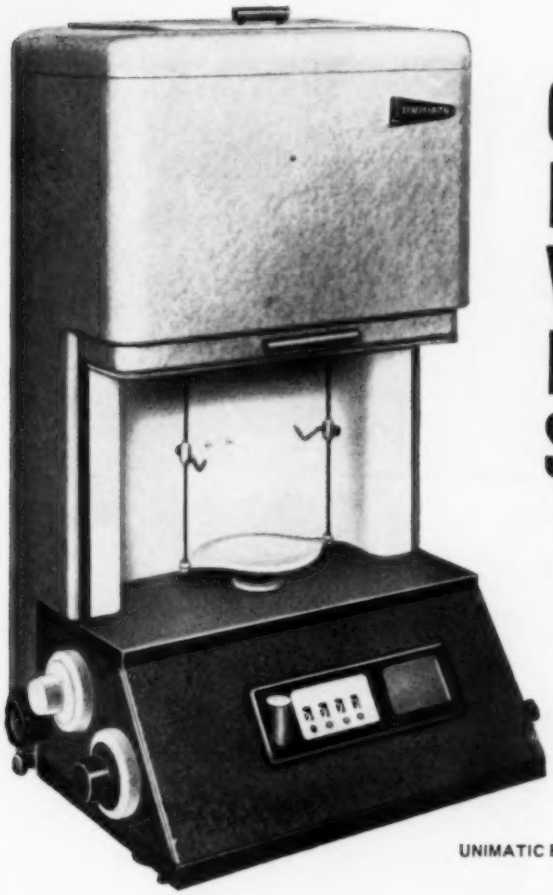

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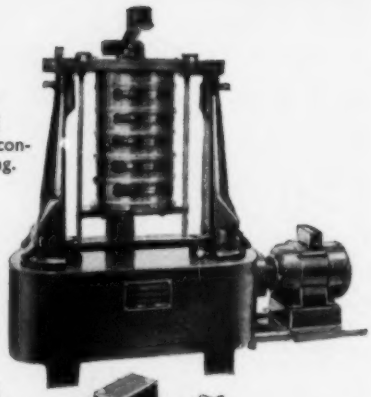

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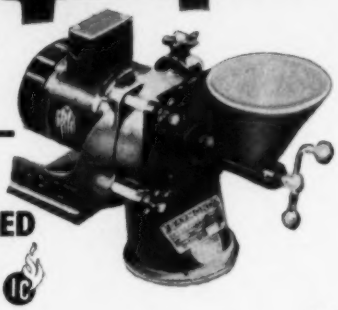

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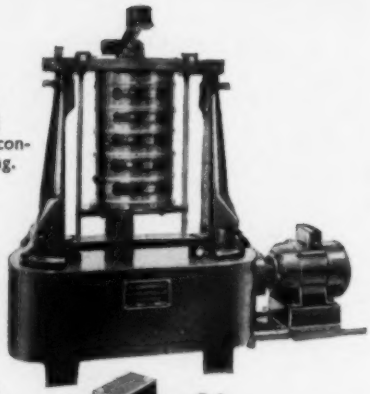

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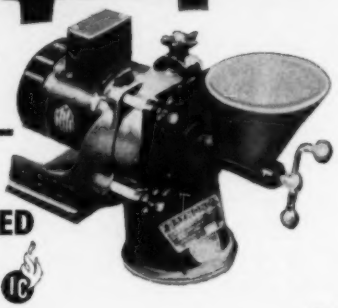

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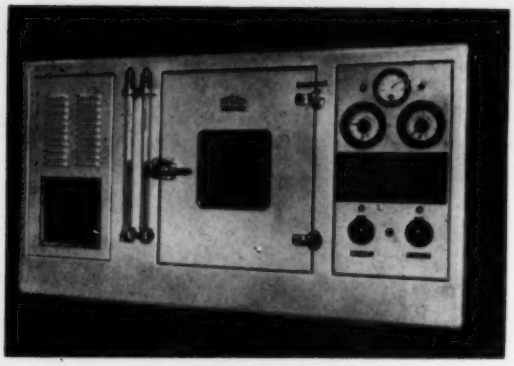

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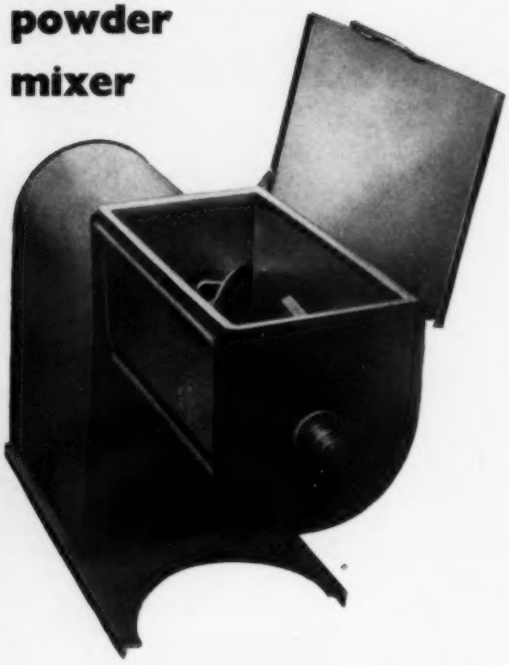

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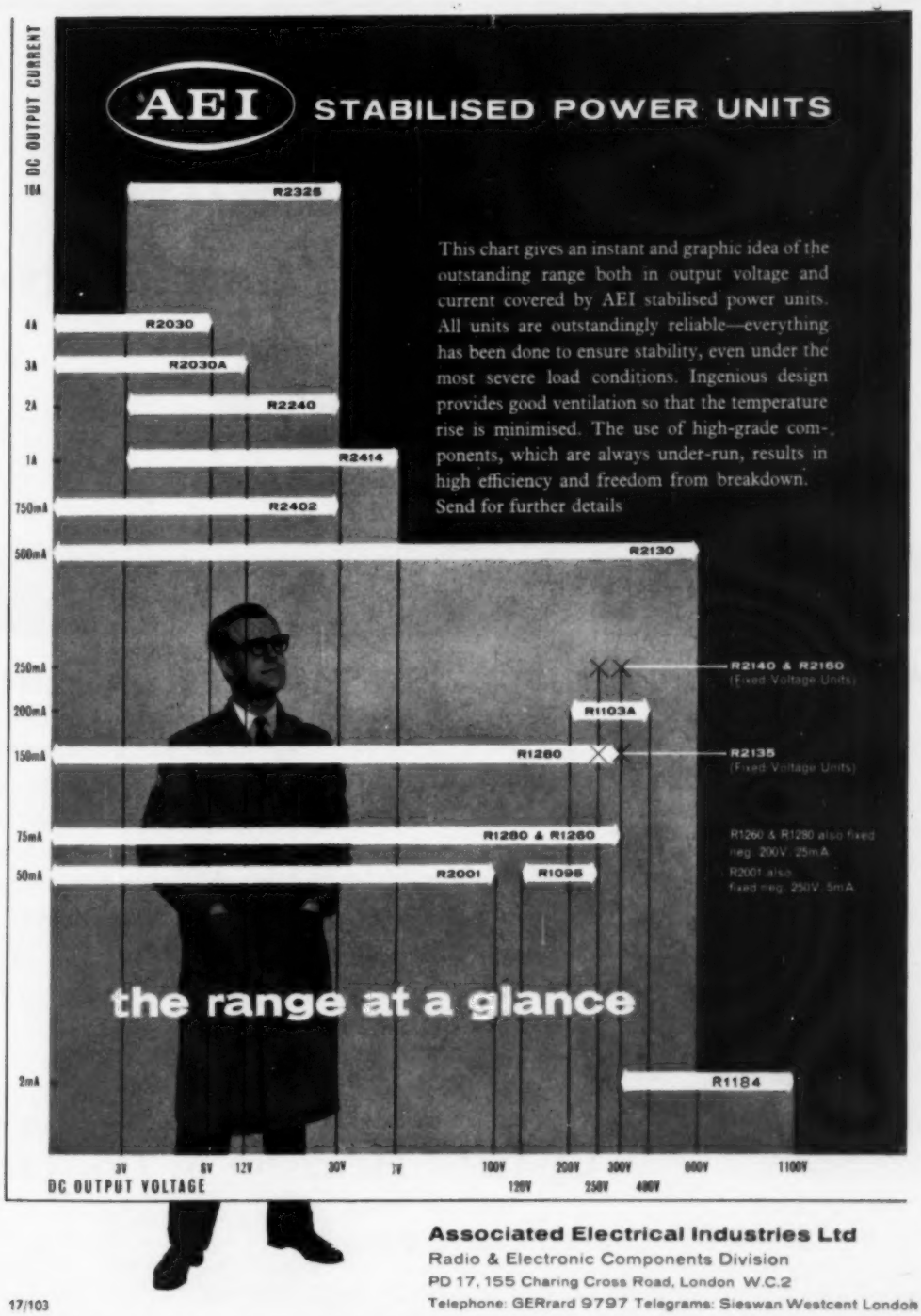

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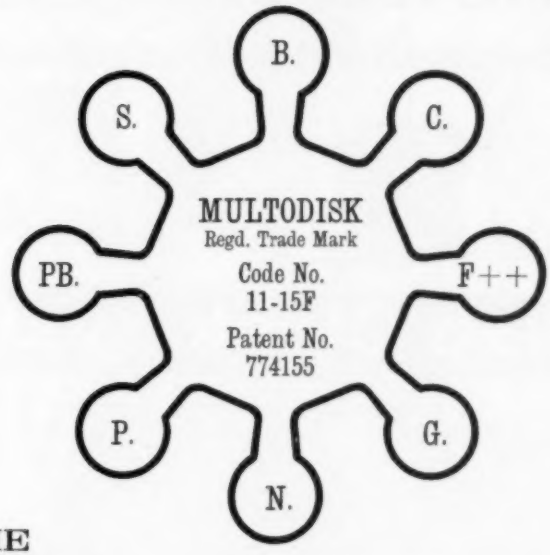

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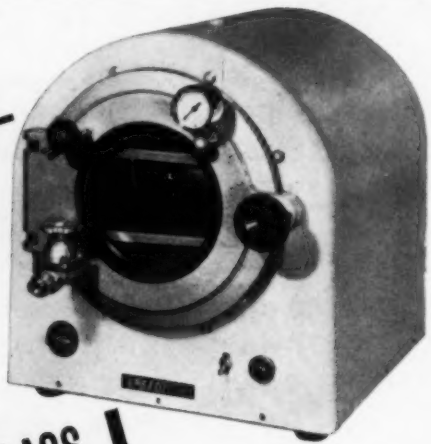

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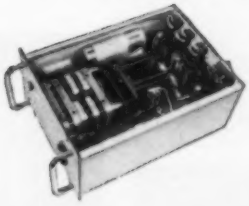

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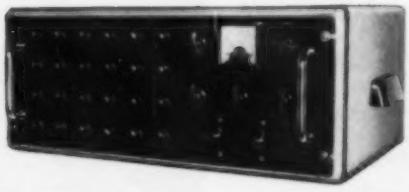

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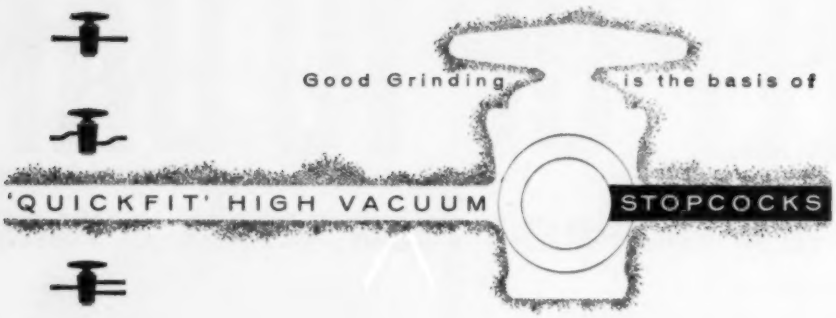

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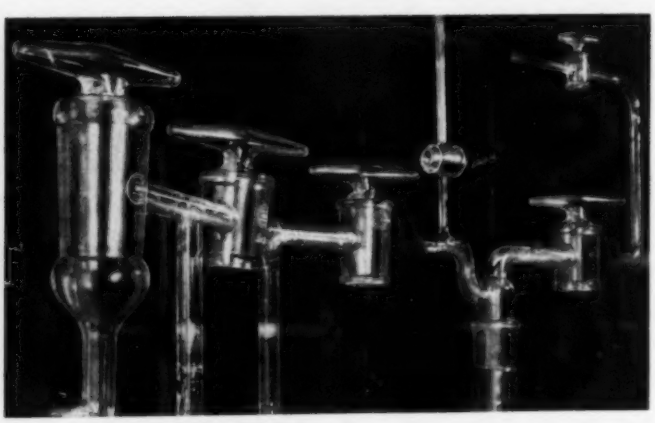

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EDITORIAL

The British Society of Rheology

IN September this society held a meeting in Llandudno to discuss rheological techniques. The President of the Society, Prof. R. W. Douglas gave his presidential address on 'The rheology of glass' and altogether 14 papers were presented by leading workers in this field. We are very pleased to have the opportunity of publishing most of these papers in this issue, which thus becomes a Special Rheological Techniques Number. The papers are introduced by Dr. Treloar, one of the best known figures in polymer physics, who was President of the Society in 1953-55 and is the author of that much-quoted book 'The physics of rubber elasticity'. This meeting was a joint one between the British Society of Rheology and the Non-Destructive Testing Group of the Institute of Physics.

Rheology is the science of deformation and flow of materials, a science which still tends to be divided into 'physics' and 'stamp-collecting' aspects. The deformation of materials is encountered in many branches of science and technology, but in very few cases are the observed phenomena amenable to simple interpretation or mathematical treatment. This is particularly true of some industrial processes. One of the objects of the founders of the old British Rheologists' Club was to provide the opportunity for workers in different fields to pool their rheological experience and thereby help each other to understand their respective observations. Such was the success of the Club that in 1950 it decided to change its name to the 'British Society of Rheology'. It now has about three hundred members.

Since the formation of the Club in 1940, a few years after the (American) Society of Rheology, several meetings have been held each year, in different parts of the U.K. In 1948, 1953, and 1958 the Society took part in International Congresses, in collaboration with societies and groups in several other countries. The British Society exchanges publications with them. In Australia, interest has been such that two formal branches of the British Society have been set up. To meet local requirements nearer home, a small informal group has recently been formed at Cambridge.

The proceedings of several of the meetings of the British Society have been published in book form. In addition, the Society issues a quarterly Bulletin; and, in collaboration with Pergamon Press, Rheology Abstracts.

A theoretician might like to meet someone with an experimental situation which is in need of analysis; an experimentalist might like to appeal to a mathematician for help. If the subject is rheological, then it is likely that a useful contact could be made through the Society. Further information about the British Society of Rheology will be gladly given to any interested person by the Hon. Secretary, Dr. M. F. Culpin, 8 The Broadway, Pontypool, Mon.

RHEOLOGICAL TECHNIQUES NUMBER

Foreword

by L. R. G. Treloar

Shirley Institute, Didsbury, Manchester 20

The papers in this issue of Laboratory Practice *were presented at a conference on Rheological Techniques held in Llandudno in September,* 1961, *under the joint auspices of The British Society of Rheology and The Institute of Physics and The Physical Society. The science of rheology, being concerned with the flow and deformation of matter, is of concern both to those who require to know the properties of materials in order to use them to the best advantage and to those whose primary interest is in trying to understand the molecular or other structural mechanisms underlying any particular type of behaviour. For either purpose, the search for more accurate and more refined methods of measurement is an essential condition for progress, and the papers which follow, though necessarily limited to a small portion of the total field, provide a good illustration of the degree of refinement in experimental techniques which is being achieved. In some cases, for example in the study of flow-birefringence, where differences of refractive index in the region of* 10^{-8} *are being measured, and in the investigation of the response of liquids to oscillating stresses with frequencies in excess of* 1,000 *megacycles per second, the available techniques are being pressed to the limit.*

Looking back over the recent developments in rheology, one is aware of a shift of attention from its more qualitative and descriptive to its more quantitative and rigorous aspects. The penalty of this evolution is that individual contributions become more specialized and therefore less easily assimilable to the general reader. This, however, is a problem in communication which is not peculiar to the science of rheology. One hopes that the presentation of these papers in Laboratory Practice *will bring readers from a wider circle into contact with a subject which in one way or another impinges on a considerable variety of experience.*

A discussion of rheological techniques clearly falls within the field of the relationship between physical and mechanical properties of materials. Although some techniques may not be adaptable to quality control, they will give information which can ultimately form the basis of a non-destructive method for control. Furthermore, physical methods of testing materials have many advantages over chemical methods. They are usually quicker, cheaper, and may not involve sampling or destruction of the material.

The Institute of Physics (now combined with the Physical Society) has long been associated with non-

(*Please turn to page 770*).

EVALUATION OF PSEUDO-PLASTIC MATERIALS BY CONE PENETROMETERS

by F. J. Mottram

Gestetner Ltd., London, N.17

Abstract

The evolution of cone penetrometer studies is traced up to the derivation of a fundamental equation for yield value by Rehbinder and Agranat. Consequences of this equation are discussed, including the significance of the exponent 'n' and its dependance upon the physico-chemical structure of the material under test. A description of the experimental procedure devotes much emphasis to the sample preparation. Collected results are given which demonstrate the degree of reproducibility of yield values using cones of different vertical angles and loadings.

A survey of some twelve homologous materials reflects a relationship in order of magnitude between cone penetrometer yield values and those obtained from flow curve data. Viscosities measured at very low shear rates (5 sec.$^{-1}$) also show a correlation with cone results.

Introduction

The cone penetrometer has been used over the last thirty years for the empirical estimation of such materials as greases and pitches. Mohr and Wellmy (1948) have successfully applied this instrument to the problem of comparing various butters but only as recently as 1949 has a sound derivation of yield value (So) from penetrometer data become available. This was the work of Rehbinder. In the equation for yield value the geometry of the cone was used to calculate a constant K, an approximate expression for which appears below.

(Apex angle = 2α)

$$K = \frac{1}{\pi} \cos^2\alpha \cot \alpha \qquad (1)$$

A more exact and lengthy expression has been derived by Agranat and Volaravich (1957) and their equation is based upon an analysis of the plastic deformation during penetration. The Agranat equation is used to calculate the values in Table I. It is interesting to note that the plastic deformation of the test material was studied experimentally by X-Ray observations during cone penetration.

The Rehbinder equation for yield value is:

$$So = \frac{K\,mg}{hn} \qquad (2)$$

Where m is the mass of the cone in gm.

g = 981 cm. sec^{-2}

h = the depth of penetration of the cone in cm.

n = is an exponent approximating to 2 but varying with the physico-chemical structure of the material under test. Consequently it can be regarded as a constant when the materials under investigation are homologous. The materials dealt with in the present investigation are basically the same in composition and method of manufacture. Thus, this exponent can be used to classify pseudo-plastic systems, in much the same way as the exponent in the de Waele-Ostwald equation.

TABLE I

2α	30°	45°	60°	90°	120°
K	0·959	0·416	0·214	0·071	0·04

Haighton (1959) by using the above analysis for yield value has met with considerable success in classifying margarines and fats with respect to their spreading properties. This amply demonstrates the advantage of using the cone penetrometer as an instrument capable of measuring fundamental physical properties.

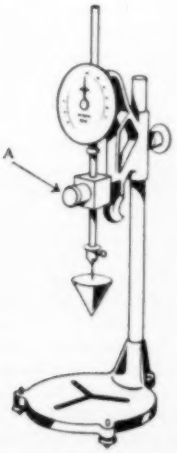


Fig. 1.

Experimental

Fig. 1 shows the general arrangment of the apparatus. The operating principle is very simple. The depth gauge is set at zero with the vertical rod pulled up to make

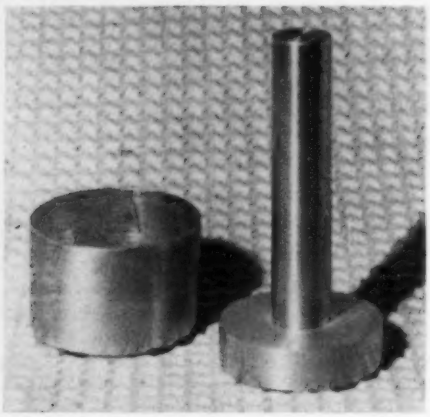


Fig. 2.

contact with the gauge. The procedure is to lower the entire assembly by slackening off the bolt A until the cone just makes contact with the test material. At this point the release knob is pressed and kept open for 90 seconds before closing; during this time the cone penetrates freely under the weight of the entire falling assembly, i.e. of the cone and the attached rod. The rod of the depth gauge can now be pushed down to make contact with the corresponding rod of the falling assembly and the depth of penetration read off directly.

Preparation of the Sample

In the A.S.T.M. publication on the cone penetrometer much space is devoted to 'working' the sample in order to ensure homogenity and expel air pockets. The remarks in this report apply chiefly to greases. With the lithographic inks investigated here working was accomplished by means of a plunger to compress the sample and a scraper to level the surface. Fig. 2 depicts the sample cup and the plunger.

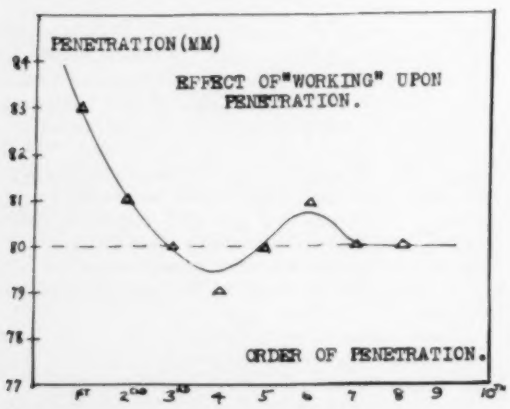


Fig. 3.

Experiment shows that constant penetration values are achieved after four or more workings (Fig. 3). The initial reading is usually biased on the high side.

Scheme of Investigation

Twelve lithographic inks were chosen for the present work. All were of conventional formulation and none of them featured a component which could be expected to place them in a different physico-chemical class from the rest.

The investigation falls into two parts:

Part A. Here the aim is to demonstrate the reproducibility of yield values using different cone angles and loadings.

Part B. This deals with the relationship between the cone yield value and other rheological expressions of rigidity, such as yield values derived from the flow curve and viscosity measured at very low rates of shear. For the former the treatment of Houwink is used. This is illustrated by the analysis of a typical pseudo plastic flow curve as in Fig. 4.

(All measurements were made at 25°C. ± 0·2°.)

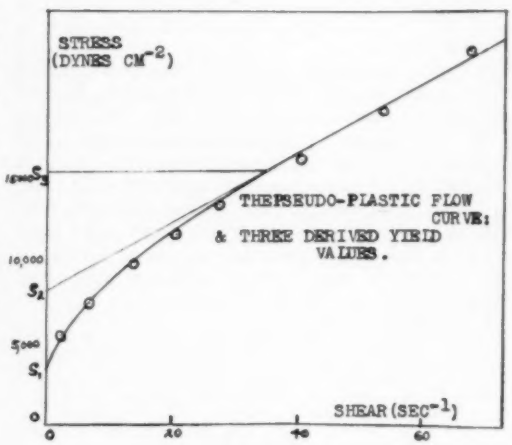


Fig. 4.

Results

The Reproducibility of Yield Values for different loadings and cone angles.

The investigation featured two inks 1183 and 1184. Their yield values were measured at two cone angles and at three different loadings for each cone. All the yield values appearing in the table below were calculated from a careful determination of h, the constant penetration (Fig. 3). For the purposes of the present investigation n (the exponent of h) was assumed to be equal to 2. The cone angles (2α) were 60° and 47° 30′ for which the values of K were respectively 0·214 and 0·360. Thus Table II is based upon the following expressions for yield value:

Where $2\alpha = 60°$ $\quad So = \dfrac{0{\cdot}214\ \text{mg.}}{h^2}$

Where $2\alpha = 47° 30'$ $\quad So = \dfrac{0{\cdot}380\ \text{mg.}}{h^2}$

TABLE II

Ink	2α	*Loading (gm.)*	*Yield Value dynes cm^{-2})*
1183	60°	55	14,200
		75	14,400
		105	15,200
	47° 30′	55	14,200
		75	14,800
		105	14,800
1184	60°	55	25,200
		75	25,000
		105	25,800
	47° 30′	55	25,500
		75	25,800
		105	25,200

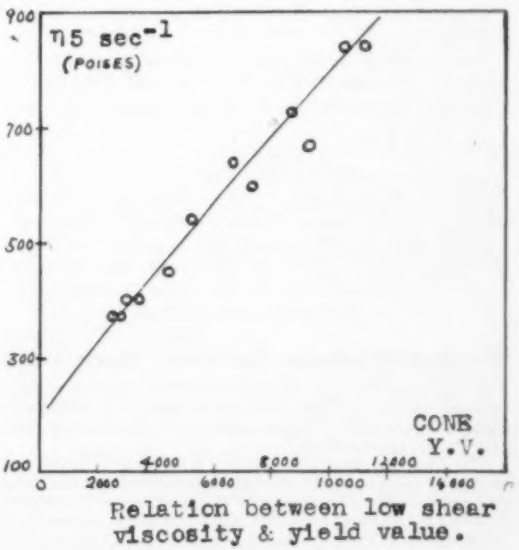
Investigation of relationship between cone yield value and other expressions of rigidity.

In the course of the work correlations were attempted between the cone penetrometer yield value and many other quantities including the three values derived from the flow curve S_1, S_2, and S_3, and also the yield values obtained from parallel plate plastometer data. Table III summarizes the investigation in that it includes only data that show or at any rate point towards a correlation with the yield value evaluated in Rehbinder's equation.

Figures for the low shear viscosity (Table III) were obtained from the flow curves used in the evaluation of S_1 for the various inks. These curves, all of which were characteristic of pseudo plastic flow, were plotted from data obtained on a cone and plate rotational viscometer.

TABLE III

Ink Reference No.	*Yield Values (dynes cm.$^{-2}$)*		*Viscosity at 5 sec.$^{-1}$ (poises)*
	Cone	S_1	
20	2,560	2,300	375
26	2,700	2,400	375
21	3,000	2,560	400
19	3,330	2,700	400
25	4,200	3,500	450
48	5,250	4,000	540
58	6,600	7,000	640
Ex. 3	7,500	6,900	690
Ex. 7	8,650	8,400	720
60	9,470	7,500	760
46	10,400	9,500	840
51	11,600	6,600	840



Relation between low shear viscosity & yield value.

Fig. 5.

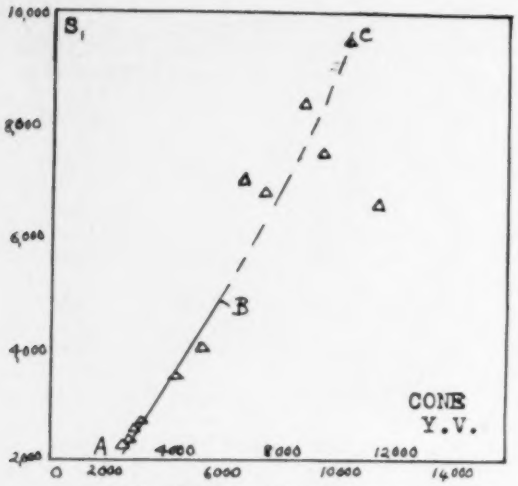


Fig. 6.

Discussion of Results

The results obtained for Part A of the present investigation indicate that cone yield values are surprisingly reproducible for two quite different cone angles and over a wide range of loadings.

In Part B of this work a quite good proportionality between viscosity at low shear rates (ca. 5 sec^{-1}) and the cone yield value is established (Fig. 5). No attempt is made at this stage to provide an explanation of this relationship.

The similarity in magnitude of the cone yield value and of S_1, up to cone values of about 6,000 dynes $cm.^{-2}$, is intriguing and will be further investigated (Fig. 6).

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Tropical Products Institutes: Open Days

For the first time the Tropical Products Institute, which became one of the DSIR's research stations in 1959, held Open Days at their London Laboratory (52-62 Gray's Inn Road) on October 19-20.

The Tropical Products Institute's work is devoted primarily to improving the economic viability of less advanced countries in the tropics especially those in the Commonwealth. It seeks to advance the technology of existing industries and the introduction of new ones as a result of research into new uses for tropical plant and animal products. Many first-class examples of this work were on view.

NEW MEMBERS OF DSIR RESEARCH COUNCIL

The Minister for Science, Lord Hailsham, has appointed five new members of the Council for Scientific and Industrial Research. They took office on October 1, 1961. They are Mr. L. H. Bedford, Director of Engineering, Guided Weapons Division, English Electric Aviation Ltd., Mr. G. B. R. Feilden, F.R.S., Managing Director, Hawker Siddeley Brush Turbines Ltd., Professor E. R. H. Jones, F.R.S., Waynflete Professor of Chemistry, Oxford University, Professor O. A. Saunders, F.R.S., Professor of Mechanical Engineering, Imperial College, London, and Mr. H. C. Tett, Chairman and Managing Director Esso Petroleum Co. Ltd. Three members who have completed their five-year term of office retired on September 30: the former Chairman, Sir Harry Jephcott, Chairman of Glaxo Laboratories Ltd., Professor C. E. H. Bawn, F.R.S., Professor of Inorganic and Physical Chemistry, Liverpool University, and Sir Willis Jackson, F.R.S., at present Director of Research and Education, A.E.I. (Manchester) Ltd. A fourth member, Sir Walter Drummond, who was also due to retire on September 30, has been reappointed for a period of three months.

Editorial

(*Continued from page* 766)

destructive testing. In its earlier stages this was mainly through the Industrial Radiology Group. In 1954 the scope of this Group was widened to include other techniques, primarily ultrasonic, eddy-current, magnetic and penetrant methods of defect detection. Its name was also changed to Non-Destructive Testing. It soon became apparent that the scope must be widened still further. Technological aspects of flaw detection, utilizing the existing and established techniques, formed the subject of discussion of other organizations but development of new techniques and the extension of existing techniques towards more stringent quality control was not otherwise being pursued. This activated the Group to ensure that its aims and objects should include the overlooking of basic aspects of quality control; to ensure that all physical principles could be brought to bear, where appropriate, on testing; to advance knowledge of relationships between physical properties and properties which must be controlled in production. This, of course, still includes a strong interest in, and share in development of, established methods of non-destructive testing.

Quality is judged not only from the absence of defects which can affect service life but also by adherence to specified properties, mechanical, physical and chemical, and by maintenance of dimensions and surface finish. Furthermore, the increasing demands for automation and the requirement for testing of all products instead of samples, requires knowledge of the inter-relations of properties of materials which is not available.

Information about the activities of the Group can be obtained from Dr. P. Lord, Royal College of Advanced Technology, Salford, 5.

DETERMINATION OF THE CONCENTRATION CHANGES OF FINE PARTICULATE SUSPENSIONS FLOWING THROUGH NARROW CAPILLARY TUBES

by H. W. Thomas

Physics Department, St. Mary's Hospital Medical School, London, W.2

Introduction

The decrease in the apparent viscosity of particulate suspensions when measurements are made in capillary instruments of narrow bore is a well-known phenomenon. Two hypotheses have been proposed to explain this. Dix and Scott Blair (1940) have suggested that, when the diameters of the suspended particles are comparable to the diameter of the capillary tube, one is no longer justified in assuming that the shear rate varies continuously across the cross-section, and that instead of the customary integration of the stress-shear rate relationship, a summation procedure should be adopted. They have derived a formula on this basis which gives a reduction in the apparent viscosity with decrease in tube diameter. On the other hand Vand (1948) and Maude and Whitmore (1956, 1958) have pointed out that the centre of a rigid particle cannot approach the tube wall to a distance closer than its effective radius, and that this results effectively in a layer of clear suspending fluid at the tube wall, and a consequent reduction in the apparent viscosity, and also in the mean concentration of the suspension within the tube. This mechanical wall effect—the Vand effect—has been shown to account adequately for the changes in viscosity and concentration observed in suspensions of plastic spheres within a narrow range of particle and tube size, and of shear stress at the tube wall. (Higginbotham, Oliver and Ward, 1958; Maude and Whitmore, 1956). There is both theoretical and experimental evidence that there is a centripetal effect arising from the presence of the tube wall over and above the Vand effect, and which is hydrodynamic in origin (see for instance Segré and Silberberg, 1961). However, a definite quantitative picture of the radial movements that can occur over the possible ranges of particle and tube diameters, of suspension concentration, and shear stress at the tube wall, is by no means available.

The author is primarily concerned with the rheology of suspensions of red blood cells, where the changes in concentration and apparent viscosity occurring in narrow capillary tubes have some physiological significance. Fahraeus (1929) seems to be the only investigator of the concentration changes of blood flowing through narrow tubes, but he reports only one series of measurements in a range of tube diameters between 50 and 1,100 μ, at one concentration (0·41) and at unspecified and probably varying values of wall stress. The method described below has therefore been developed in order to obtain a more complete picture of the concentration changes that can occur with red cell suspensions over as wide a range of conditions as possible. It is proposed to correlate all the results with a parallel series of viscosity measurements on similar suspensions. The nett inward drift of the particles of the suspension when flowing through the tube results in a reduction in both the concentration and the apparent viscosity. The Dix-Scott Blair 'sigma' effect can only influence the apparent viscosity. Hence the proposed correlation, if sufficient accuracy is attainable, should enable one to decide under what conditions the shear rate-shear stress relationship breaks down as the particle size approaches the tube diameter. A suitable method of analysis has been developed for this purpose.

Experimental Method

Maude and Whitmore (1956) determined the concentration of suspensions of plastic spheres (90-100 μ in diameter) flowing in a tube nearly 2 mm. in diameter by trapping the suspension within a 60 cm. length, and determining the dry weight of the entrapped spheres. Clearly, such a method was out of the question for blood in tube diameters of the order of 500 μ and below which are necessary to get observable effects. Fahraeus (1929) trapped the blood within 5 cm. lengths of capillary tubes, and centrifuged the tubes at unspecified conditions. It is known that a correction has to be made for the suspending fluid trapped within the packed sediment, and with red cell suspensions the author has found by direct determination that 2 per cent of the red cell column is suspending fluid even after two hours centrifugation at an effective acceleration of 3,000 g. in standard haematocrit tubes which have an internal bore of about 3 mm. This correction for the trapped supernatant varies with the conditions of centrifugation, and it was anticipated that it would be extremely difficult to get a suitable small and consistent correction to apply to results obtained in capillary tubes with bore diameters well below 1 mm.

In principle, the concentration of a suspension can be determined by labelling the suspending phase with a radioactive isotope and assaying the relative amounts of the activity in equal volumes of the suspension and the suspending fluid. It remained to find out if this method could be applied to the determination of the concentration of red cells within narrow bore capillaries, and in particular if reasonable amounts of the isotope would give the desired degree of accuracy.

Hitherto suspensions of human red cells in standard

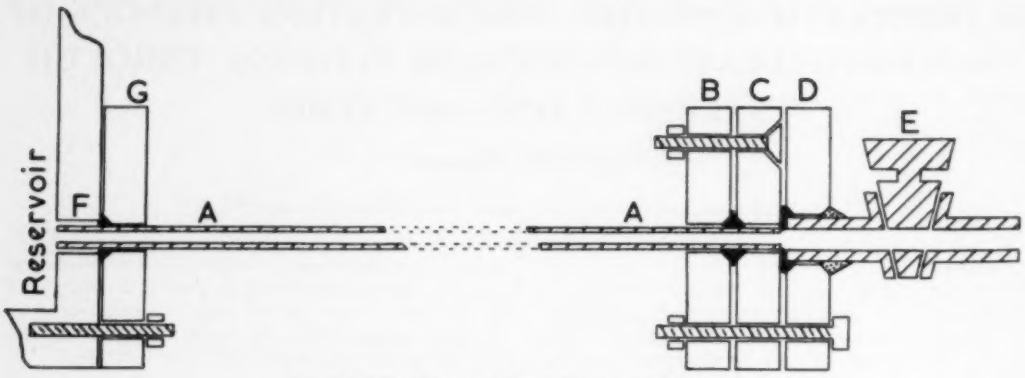

Fig. 1. Sketch diagram of the capillary tube fixture.

A.C.D. (acid citrate dextrose) solutions have been used. The suspending solution has been labelled with radio-iodinated human serum albumen which is known not to enter the red cell, is readily available (Radiochemical Centre, Amersham) and provides a convenient γ-emission for radioactive assay of the suspension concentrations. The capillary tubes were obtained commercially (Chance) and calibrated by means of the mercury method. The method of mounting the capillary tube is illustrated in Fig. 1. The two Perspex discs B and C, with a rubber 'O' ring in between, form an end-plate for the capillary tube A. The disc D is cemented with Araldite to form a similar end plate for the ground glass tap E. By means of three 4 B.A. bolts the tap may therefore be readily attached or separated from the capillary tube. By means of the Perspex disc G engaging another 'O' ring, the other end of the capillary tube is held within the porthole F such that its end is flush with the inside surface of the reservoir. This provides a seal which has been found adequate for excess pressures up to 1 atmosphere.

The suspension in the reservoir is maintained uniformly mixed by means of a magnetic stirrer. Air pressure is applied to the reservoir and when the flow is sufficiently established, the tap is closed. The pressure is released, the whole apparatus tilted through about 30 degrees so that the porthole F is raised clear of the suspension, and the capillary tube and its contents removed. Since the bore of the tap is in general much larger than that of the capillary, and therefore contains a large amount of radioactivity, it is separated from the capillary tube. The ends of the capillary tube are sealed ('Parafilm' sheet has been found convenient for this purpose) and the radioactivity contained within a certain length determined. For the small bore tubes of diameters 100 μ and below, the flow rate is generally exceedingly low, and it has been found necessary to fill the capillary tube with the suspending fluid before affixing to the reservoir, so that incompressible liquid and not air is trapped on the reservoir side of the tap on closure.

Fig. 2 is a sectional diagram of the arrangement for the determination of the radioactivity. Two conventional sodium iodide scintillation counters together with the cylindrical lead shielding A are held in position by grub screws fixed through the brass cylinder B. Vertical slots are cut in both the lead shielding A and the brass cylinder B down to the level PP ($\frac{1}{8}$ in. below the centre line 00) so that the capillary tube can be placed in a horizontal position between the surfaces of the two crystals. A cylinder of lead C, split at d to allow the change of capillary tubes, and with a cut away opposite the position of the capillary tube, provides extra radiation shielding. The bottom of the slot in the brass cylinder is cut at 60 degrees to the vertical, so that the capillary takes up a fixed position. A mark on the capillary tube is aligned with a mark on the brass cylinder, and hence the position is strictly fixed and reproducible. The reproducibility of the counting geometry has been checked directly, and found to be better than $\frac{1}{2}$ per cent. No significant changes in counting rate due to sedimentation of the suspensions have been observed within the normal periods of counting in tubes up to 1 mm. in diameter.

The voltage pulses from the two counters are added at the input of the linear amplifier, and counted by means of a scaler (which counts all pulses of a size greater than a predetermined voltage peak). Sufficiently large counts are taken to achieve the desired degree of accuracy.

When the trapped suspension has been counted, the capillary tube is washed clean, dried, and checked in the counter set-up to make sure that there is no activity remaining on its walls. The tube is then filled with clear suspending fluid obtained by centrifugation of some of the labelled suspension, and the activity determined under the exact conditions used when the tube contained the suspension. The ratio of the two counts gives a value for the concentration of suspending fluid in the suspension, and the value for the concentration of red cells is obtained by subtraction from unity. As described the method would therefore be extremely inaccurate

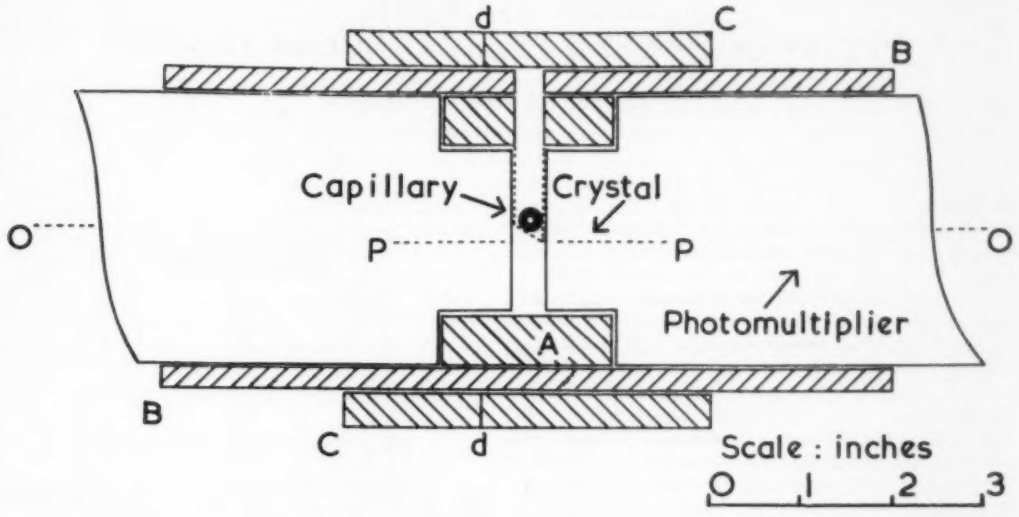

Fig. 2. Diagram to illustrate the counter arrangement.

for studies of dilute suspensions; for example, at a red cell concentration of 0·1, a 1 per cent error in the ratio of counts results in a 10 per cent error in the determined concentration. Under such circumstances it would be advisable to label the red cells with a suitable isotope (^{51}Cr suggests itself) and to compare the count obtained from the suspension with that obtained from a solution of lysed cells of a known concentration.

Results and Evaluation

The few preliminary measurements carried out with the apparatus have demonstrated the significant decrease in red cell concentration with tube diameter. For a suspension concentration of 0·37 in the reservoir, and at a calculated wall stress of 100 dynes per square centimetre, the concentration has dropped by about 8 per cent in a tube of diameter 200 μ.

The limitation to the method is the considerable counting times required to obtain reasonably low errors (due to the statistical variation of counting rates) in the case of the smaller bore tubes. The background counting rate for the described set-up is just under 1,000 counts per minute. With a capillary tube filled with a solution labelled with ^{131}I isotope, the source count above background s (counts per minute) is given approximately by the relation $s = 0{\cdot}022\ c\ d^2$, where c is the activity in the suspension expressed in microcurie per millilitre, and d is the diameter of the capillary tube expressed in microns. Thus with an activity of about 20 microcurie per millilitre (1 millicurie in 50 millilitre of suspension contained in the reservoir) the source counting rate for a 100 μ bore tube is only 4,400 counts per minute above the background of about 1,000 counts per minute. Counting times of the order of 20 minutes are then required to limit the statistical error of each radioactivity determination to about 0·5 per cent. With smaller tubes the long counting periods necessary become a serious factor in the conduct of the experiment, and the replacement of the present scaler by a single channel pulse height analyzer is to be recommended. This instrument, by counting voltage pulses within a narrow band of pulse heights corresponding to the narrow energy band of the strong γ-emission of the ^{131}I isotope, would give a far better discrimination between the source and the background counts. In conclusion, one cannot but confess that the method is tedious, but it can at least provide valuable experimental data that have been extremely difficult to obtain by any other technique.

ACKNOWLEDGMENT.—*The work described has been carried out during the tenure of a Leverhulme Research Fellowship. The author wishes to acknowledge his indebtedness to Dr. S. Rowlands and Dr. A. C. Groom for many valuable discussions, and to Mr. M. D. G. Thomasse and Mr. J. H. Baker-Self for technical assistance.*

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SOME ACOUSTIC TECHNIQUES USED IN MOLECULAR PHYSICS

by R. Holmes and W. Tempest

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Abstract

An ultrasonic pulse transmission method and an audio-frequency tube resonance method for the measurement of sound velocity and absorption in gases are described. A brief account is given of the use of the sound propagation constants obtained from the measurements in determining the factors affecting energy exchange in intermolecular collisions.

Introduction

The mechanism of energy exchange between the translational and the internal motion of gas molecules is of considerable interest to the molecular physicist. One method of studying this effect is by measuring sound velocity and absorption as a function of frequency and pressure. The time lag for the energy exchange results in a velocity dispersion and a peak in absorption at some frequency/pressure (f/p) ratio. This f/p ratio has been found to vary over wide limits. For example, for the vibrational-translational energy exchange in oxygen it is at about 50 c/s atm. whereas for the rotational-translational energy exchange in nitrogen it is at about 400 Mc/s. atm. Two sets of apparatus have been developed in this laboratory for the study of both sound velocity and absorption. The first is a pulse transmission method useful in the f/p range 50 kc/s. atm. to 40 Mc/s atm., and the second is an audio-frequency tube resonance method useful in the f/p range 30 c/s. atm. to 3 kc/s. atm.

Pulse Transmission Method

Introduction.—Fig. 1 shows a schematic diagram of the apparatus used in the pulse transmission method. It consists essentially of two transducers mounted in a gas tight vessel, and the associated electrical system.

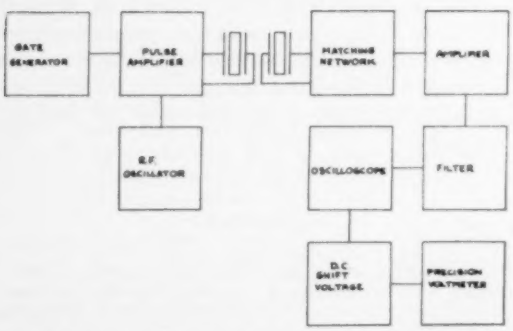

Fig. 1. Schematic diagram of pulse transmission method.

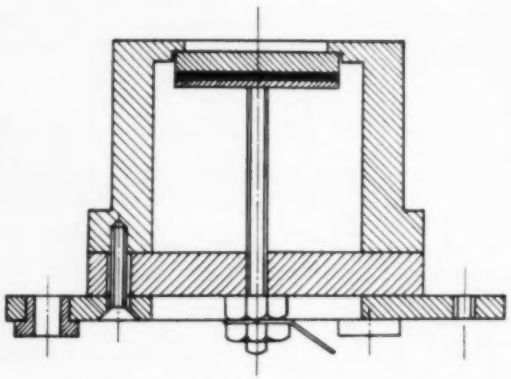

Fig. 2. Cross-section of crystal transducer.

The two transducers are mounted opposite each other, one fixed and the other on the end of a piston. The piston moves in an accurately machined bore and when the transducers have been set parallel they remain parallel within one minute of arc for all separations. A travelling microscope screw thread mounted in the end of the piston and running through a split nut permits the transducer separation to be varied from 1/2 mm. to 60 mm. The pitch of the screw is 1mm. and the transducer position can be determined to 1/200 mm.

Crystal Technique.—For frequencies between 400 kc/s. and 3·5 Mc/s. the transducers are X-cut quartz or barium titanate crystals operated in thickness mode resonance. A block of polythene is cemented to the back face of each transducer to reduce the 'Q' to about 15 so that high frequency electrical pulses can be transmitted. Fig. 2 shows a typical crystal mounted in its holder. The earthed metal of the holder helps to reduce electrical crosstalk between the transducers. Polythene sheet between the holders and their supports reduces to negligible proportions the mechanical cross talk travelling through the walls of the gas vessel.

Rectangular radio-frequency pulses are produced by modulating the output of a stable oscillator. The modulating pulses are variable in length from 8-50 μs and the pulse repetition frequency is 300 cycles per second. The modulator output is amplified and pulses of 800 volts r.m.s. amplitude are applied to the transmitter transducer.

The receiver-transducer output is matched to the input of a stable gain, wideband amplifier (86 db. maximum gain), filtered to reduce hum and noise, and

displayed on a D.C. oscilloscope. All H.T. and L.T. supplies are stabilized so that the pulse amplitude shows no detectable change over a one hour period.

Condenser Technique.—At frequencies below 400 kc/s. the use of crystal transducers presents some difficulties. The electrical input impedance increases and the diameter must be increased to prevent excessive divergence from a parallel beam of sound.

To avoid these difficulties, condenser transducers are used. (Fig. 3). These consist essentially of a polished metal plate over which is stretched a sheet of aluminized 'Melinex' 25 microns thick with the conducting face away from the metal backing plate. The front plate is earthed and the live backing plate is screened by the earthed holder. These transducers are aperiodic and can be operated up to 500 kc/s. In operation both receiver and transmitter are polarized with 300 volts D.C.

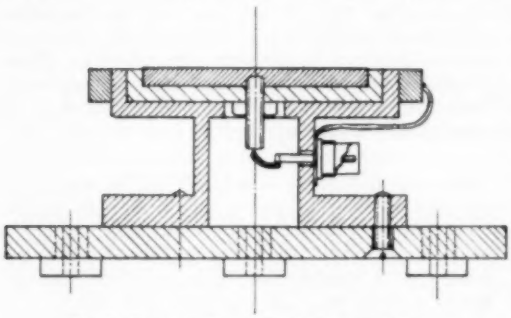

Fig. 3. Cross-section of condenser transducer.

The transmitter transducer is driven by a radio-frequency power amplifier giving 140 μs pulses of rectangular envelope at 100 pulses per second. The radio frequency drive for the power amplifier is derived from a continuously running crystal oscillator. The receiver transducer output is amplified as with the crystal transducers and displayed on a D.C. oscilloscope.

The condenser technique was developed in this laboratory by Dr. H. D. Parbrook.

Temperature Control and Gas Handling.—The gas vessel is immersed in a water bath (for the range 25 °C. to 80 °C.) or a water-glycol bath (for the range −10 °C. to 25 °C.). The bath is maintained within ± 0·1 °C. of the required temperature by means of an immersion heater and thermostat.

The gas vessel is connected to a gas handling system and a two stage vacuum rotary pump so that it can be evacuated and filled with the test gas.

Sound Propagation Measurements.—Sound absorption is determined by measuring the amplitude of the received pulse as a function of the transducer separation. The pulse amplitude is measured by adding a D.C. voltage sufficient to displace the pulse through its own height, the D.C. voltage being measured on a 'Precision' grade meter. The assumptions made are that the D.C. voltage is proportional to the amplitude of the pulse and that there is a linear relationship between the received signal and the transducer output. These assumptions are verified experimentally.

Sound velocity is measured by comparing the phase of the received pulse with the radio-frequency output through Lissajous figures on an oscilloscope. The transducer displacement required for a complete cycle of the ellipse is one wavelength. The radio-frequency output is monitored on a Racal SA21B frequency meter and hence the velocity is found from $V = f\lambda$.

Accuracy of Sound Propagation Measurements.—The possible error in an individual absorption measurement depends mainly on the gain stability of the amplifiers, the systematic error in the voltmeter, the random error in the determination of the slope of the log pulse height versus distance graph and the error due to non-planar sound field. The maximum possible error is estimated to be 2-3 per cent when the absorption is moderately high ($\alpha\lambda > 0{\cdot}05$) but may increase to 5 per cent for low absorption ($\alpha\lambda = 0{\cdot}02$-$0{\cdot}01$). These figures apply up to 10 Mc/s. atm. Above this there is a rapid loss of signal with decreasing pressure so that the error increases to 5-10 per cent as f/p increases from 10 to 40 Mc/s. atm.

The possible error in an individual velocity measurement depends on temperature stability, frequency stability of the oscillator, wavelength determination and the departure of the sound field from plane wave propagation. The maximum possible error is estimated to be 1-2 parts per 1,000 up to 10 Mc/s. atm., increasing to 5 parts per 1,000 at 40 Mc/s. atm.

The Resonator Tube Method

Introduction.—Fig. 4 shows a schematic diagram of the apparatus used in the resonator tube experiments. The method of operation is as follows. The a/f oscillator is tuned as closely as possible to a resonant mode frequency of the tube, while the automatic changeover switch connects the transducer alternately to the

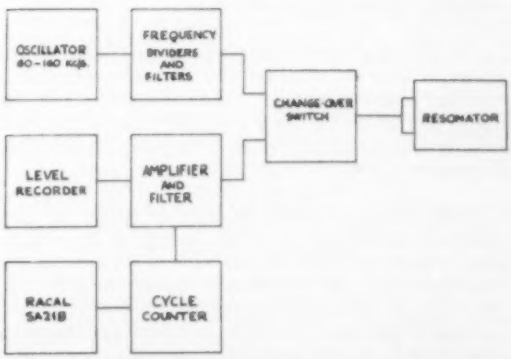

Fig. 4. Schematic diagram of resonance tube method.

oscillator and the amplifier for periods of four seconds. During the first period the transducer drives the resonator and the amplitude of the oscillations in the resonator builds up to a steady value; when the switch operates the transducer is connected to the amplifier and serves as a sound receiver producing a signal corresponding to the exponential decay of sound in the resonator. The frequency of this decaying exponential is measured by the cycle counting circuit and a Racal SA21B microsecond timer; the level recorder (B and K type 2304) records the rate of decay.

The Resonator Tubes.—Two resonator tubes, of 359 and 61 cm. length respectively, are used to cover the frequency range from 180 c/s. to 1,500 c/s.; as these tubes are of similar design it will be sufficient to describe only the larger of them. This is a cylindrical steel tube of 30 cm. inside diameter with a 0·6 cm. thick wall; flat brass plates are fitted into the ends, sealing being achieved by means of rubber 'O'-rings. The transducer is a commercial pressure unit (of the type used in 15 watt horn loudspeakers) mounted on the outside of the endplate, and coupled to the resonator by a hole of 1·2 mm. diameter at the centre of the plate. The necessary electrical leads to the transducer are brought out through glass-metal seals in a brass 'hat' which covers the transducer. The resonator can be evacuated and filled with gas through three holes, of 1·2 mm. diameter, in the other endplate.

Temperature Control.—The velocity of sound in a gas varies as the square root of the absolute temperature and since the apparatus is designed to make velocity measurements to a precision of 0·01 per cent, a temperature control to about $\pm$ 0·03°C. at 30°C. is needed. To attain this control the resonator tubes are mounted in a water tank of 350 gal. capacity, the water being continuously circulated at the rate of 30 gal./minute through a 20 gal. tank containing an immersion heater. A thermistor bridge thermostat, with a differential of 0·03°C., is mounted in the main tank and controls the immersion heater. It is found that by this means the required temperature stability can be reliably maintained for runs taking several days.

Gas Filling System.—A two stage rotary vacuum pump is used to evacuate the resonator tubes, the ultimate pressure being 10^{-2} mm. of mercury after 36 hours pumping. The long pumping time is required because of the small diameter of the holes coupling the large tube to the vacuum system.

Audio-frequency Oscillator.—Some modes of the resonator tubes have 'Q' values as high as 500 and to excite these resonances an oscillator of good frequency stability with fine incremental tuning is required. Several commercial oscillators were tested but none proved satisfactory. A suitable oscillator was therefore constructed of the inductance capacity type, with variable capacity tuning and operating in the range 80 to 160 kc/s. The oscillator output is shaped and fed into a chain of 12 bistable frequency dividers thus providing outputs at octave intervals below the oscillator frequency down to a minimum of 20 c/s.

Amplifier, Filter, and Frequency Measuring Unit.—The amplifier and filter unit is a B and K type 2105 which incorporates a continuously tunable filter with a band width adjustable between 1/6 and 1/40 octave.

The filter output is fed into a 'cycle counting unit' which converts the sinusoidal signal into a pulse form which is then fed into a counting circuit giving one output pulse for 64 or 128 input pulses. These output pulses are used to operate a Racal SA21B frequency meter which times the interval between pulses to an accuracy of $\pm$ 1 μs. By this means the frequency of the received signal can be determined to an accuracy of $\pm$ 0·005 per cent.

To measure the rate of decay of the resonator oscillations, the filter output is fed into a B and K level recorder with a logarithmic characteristic which provides a plot of log signal amplitude against time. From this the rate of decay can be determined to an accuracy of $\pm$ 3 per cent.

Treatment of Results.—The values for the frequencies and rates of decay of the modes yield values for the sound velocity and absorption in the tube. To relate these to the 'free-space' propagation constants the results are corrected for the effects of viscous drag and thermal conduction to the tube walls and ends.

Accuracy of Sound Propagation Measurements.—The absolute accuracy of the velocity measurements is estimated to be $\pm$ 0·07 per cent, while the relative accuracy of a series of measurements in the same gas is about $\pm$ 0·03 per cent.

Absorption coefficients can be obtained to an accuracy within $\pm$ 5 per cent.

Uses of Sound Propagation Measurements

The principal use of sound propagation measurements is to evaluate the relaxation times for energy transfer between translational and vibrational motion and between translational and rotational motion of molecules.

If the simplest assumption is made that the energy exchange is governed by a single relaxation time then the sound velocity may be written as,

$$V^2 = \frac{RT}{M} \frac{C_{po}C_{vo} + 4\pi^2(f/p)^2K^2C_{p\infty}C_{v\infty}}{C_{vo}^2 + 4\pi^2(f/p)^2K^2C_{v\infty}^2}$$

and the absorption per wavelength as,

$$\alpha\lambda = \frac{\pi RC^1\, 2\pi(f/p)K}{C_{po}C_{vo} + 4\pi^2(f/p)^2K^2C_{p\infty}C_{v\infty}}$$

where, T = absolute temperature
M = gramme molecular weight
R = gas constant for one gramme molecule
f = sound frequency
p = gas pressure
K = relaxation time at unit pressure
C_{vo} = molar specific heat at constant volume $(2\pi(f/p)K \ll 1)$
C_{po} = molar specific heat at constant pressure $(2\pi(f/p)K \ll 1)$
$C_{v\infty}$ = molar specific heat at constant volume $(2\pi(f/p)K \gg 1)$
$C_{p\infty}$ = molar specific heat at constant pressure $(2\pi(f/p)K \gg 1)$
C^1 = relaxing specific heat

and

$$C_{vo} = C_{v\infty} + C^1 \quad C_{po} = C_{p\infty} + C^1 \quad C_p = C_v + R$$

Hence the measurement of the whole of the velocity dispersion or of the absorption peak will yield values for both C^1 and K. If only a portion of the curves can be measured K can still be found if C^1 is known from spectroscopic data.

The relaxation time K is related to the number of first excited state to ground state transitions per molecule per second, k_{10}, by the equation,

$$1/K = k_{10}[1 - \exp(-h\nu/kT)]$$

where ν = fundamental vibrational (or rotational) frequency.

It is generally assumed that transitions only occur in binary collisions in gases so that if N is the total number of collisions per molecule per second at unit pressure the average number of collisions necessary for de-excitation, Z, is N/k_{10}. The probability of de-excitation per collision for an excited molecule P_{10}, is $1/Z$.

For a more thorough account of the theories of translational-vibrational and translational-rotational energy exchange see Herzfeld and Litovitz (1959).

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D.S.I.R. ROAD RESEARCH TEAM HELPS THE U.S.A.

A British research team from the D.S.I.R.'s Road Research Laboratory has been asked by U.S.A. authorities to make skidding resistance studies on an American runway. The party, which left London recently, went at the request of the National Aeronautics and Space Administration and the Federal Aviation Agency, U.S.A., who are financing the project. With their specially designed high speed trailer towed by a Jaguar XK-150 they will make high speed tests on an airfield at Atlantic City, New Jersey.

The Road Research Laboratory is the only organization in the world to have developed this particular technique of testing skidding resistance at high speed.

The three-man team is led by Mr. J. Masters, who is in charge of the field trials. He is accompanied by Mr. K. H. F. Cardew, who was largely responsible for developing the apparatus and Mr. J. K. Meads, an expert driver, who will drive the Jaguar during testing.

Giant Computer for London University

To Accelerate Pace of Britains Scientific Research

A great acceleration in the pace of Britain's scientific research will become possible with the installation at the University of London of a giant new computer—the Ferranti Atlas—towards the end of 1963 at a cost of approximately £2M. This machine is claimed to be the most powerful computer in the world. Computers of this size are urgently needed by British universities to meet the ever increasing volume of computation involved in present-day research projects.

The work which Atlas will perform at the University of London will range over many fields of science and engineering and will be far wider in scope than that possible with the present Mercury computer. In some respects the Atlas will continue with the same type of computations as are done on Mercury, but will be able to handle a much greater volume of work.

One example of this is in crystallography. Calculations on crystal structure already account for a major part of the computing done on Mercury, and work has already begun on the larger molecules such as proteins and the nucleic acids. London has the largest body of crystallographers in the country. There are active groups at Birkbeck, King's and University Colleges, at the Royal Institution and the National Institute for Medical Research. These, with a new group which has recently been set up at Imperial College, undoubtedly need much greater facilities for computation than can be provided at present. Computational methods in crystallography are still in their infancy and will be greatly developed in the future.

The Atlas will also make possible extensive calculations on mechanical structures. Very large matrices arise in the study of aircraft and civil engineering structures, while complex non-linear problems occur in the investigation of creep, plasticity and heat transfer effects.

Computing techniques are already being used to some extent in weather forecasting, but on the research side of meteorology a most fruitful application for such a powerful machine as the Atlas will be to the numerical study of mathematical models of the atmosphere.

Partial differential equations are very commonly used in engineering and other sciences to express dynamic conditions, but their solution by numerical techniques often presents great difficulties. The universities have hardly tackled this field of work because of the lack of suitable computers. But there are important applications to electro-magnetic wave propagation, fluid dynamics, stellar structure and chemical engineering, for all of which a large, fast computer is essential.

Now that artificial satellites are beginning to appear in the skies in such profusion, a great many records from these devices will be arriving for analysis by the Space Research Group at University College. Reduction of these will present an enormous computing problem which will be within the capacity only of large machines like the Atlas.

Linguistic analysis and mechanical translation are rapidly growing fields of investigation at the University of London, and require a computer with a large storage capacity. Analysis of non-numerical information, using a suitably designed information processing language, is also a computing problem of exceptional interest. With the aid of Atlas it will be possible to develop and apply advanced techniques in this field, for example in the construction of autocodes, the heuristic solution of logistic problems and in the simulation of psychological processes such as learning.

MEASUREMENT OF DOUBLE REFRACTION IN LIQUIDS PRODUCED BY ULTRASONIC RADIATION

by N. C. Hilyard, B.Sc., Grad.Inst.P

Department of Physics, University of Southampton

Abstract

When a liquid or solution is irradiated with acoustic waves it becomes optically anisotropic and behaves as a uniaxial crystal with its optic axis lying along the direction of propagation of the wave. This effect has been observed in liquids of different viscosities including polymer solutions, which show a small birefringence. Measurement of the double refraction induced in the solution gives information on molecular parameters in much the same way as flow double refraction. The apparatus used in this study, which employs a visual method of measuring the double refraction, is described and the accuracy that can be obtained is discussed.

Introduction

When an ultrasonic wave is passed through a pure liquid or solution of large molecules the molecules are orientated and the medium becomes optically anisotropic, the optic axis lying along the direction of propagation of the sound wave. The actual mechanism of the orientation depends on the size of the molecule. With small molecules the orientation is attributed to the velocity gradient set up in the liquid, whereas with large rigid molecules it is supposedly caused by radiation pressure. If linearly polarized light is passed through the medium with vibration direction at 45° to the optic axis it emerges with a phase difference $\Delta = \frac{2\pi d \Delta n}{\lambda}$ between the two linearly polarized components. Here, Δn is the difference in refractive indices in directions parallel and perpendicular to the optic axis and normal to the incident light. The path length through the medium is d, and λ is the wavelength of the incident light *in vacuo*.

This effect, known as acoustic double refraction, is intimately connected with flow double refraction, and Lucas (1938, 1939) who was the first to observe it, gave an explanation in terms of early hydrodynamic theories for flow double refraction. Peterlin (1950a, 1950b) assuming an ellipsoidal model for the molecule has shown that the birefringence in a pure liquid induced by a plane progressive sound wave travelling in the y direction is related to the acoustic intensity $\bar{\omega}$ by

$$\Delta n = f\{n, (\alpha_1 - \alpha_2), b, \omega, c, \rho, v, \tau, (\omega)^{\frac{1}{2}}\} \times \sin\{\omega(t - y/v) - \varnothing\} \quad \ldots (1)$$

where the phase angle $\varnothing = \tan^{-1}(\omega\tau)$. In this equation, n is the refractive index of the medium, $(\alpha_1 - \alpha_2)$ is the optical anisotropy, b is a function of the axial ratio of the ellipsoidal molecule, ω is the angular frequency of the acoustic wave, c is the concentration, ρ is the density of the medium, v is the velocity of sound in the medium, and τ is the relaxation time of the molecule.

As the effect is very small (Δn is of the order 10^{-7} to 10^{-9}) except in the more viscous liquids, few experimental data are available to verify the theories. However it has been shown by Badoz (1957) and Zvetkov Mindlina and Makarov (1946) that the double refraction in pure liquids varies according to the square root of the intensity as predicted by equation (1). Solutions of deformable large molecules behave in much the same way as pure liquids but the effect is much smaller.

Design Factors

Considerations of the design of the apparatus fall into two distinct groups. They are (1) the optical system, and (2) the ultrasonic system, which includes the transducer and the test cell.

Optical System.—From equation (1) it can be seen that the double refraction varies with time and consequently cannot be compensated by normal methods. The requisites for a satisfactory optical system are a suitably polarized parallel beam of light passing through the anisotropic liquid normal to the direction of propagation of the acoustic wave, and a means of analyzing the emerging elliptically polarized light.

A suitable method of measuring the double refraction has been devised by Zvetkov (*loc. cit.*) who introduced a Wollaston double image prism between the cell and the analyzer. When natural light is incident upon this prism two divergent beams of light emerge polarized at right angles. If we call the direction of propagation of the acoustic beam azimuth zero, the polarizing prism is set at 45°. The Wollaston is set so that its vibration directions are parallel and perpendicular to the vibration direction of the polarizer. When the analyzer is crossed with the polarizer there should be no light flux emerging from it, but when the medium is anisotropic one image becomes brighter. The angle β through which the analyzer must be rotated to make the two images of equal intensity is related to the r.m.s. birefringence (Δn) by

$$\tan\beta = \frac{\pi d}{\lambda}(\Delta n) \quad \ldots\ldots\ldots (2)$$

The sensitivity is limited by the smallest intensity difference which can be detected by the eye and the accuracy to which the angle of rotation can be measured.

It is found that complete extinction can not be

obtained, due to the strain birefringence in the windows and depolarization by the liquid and the Wollaston prism. Also a third undeviated image is produced due to the vibration directions of the two wedges comprising the Wollaston prism not being exactly at right angles. Initially the intensity of the two deviated images are made equal by rotating the analyzer. In the present arrangement the need for mechanical rotation of the analyzer is overcome by introducing a Faraday coil. The Faraday coil merely rotates the vibration direction of the polarized light, and is thus equivalent to a rotation of the analyzer. The rotation it produces is proportional to the current through the coil, which can be measured accurately.

The inclination and divergence of the light beam has a considerable effect on the measured value of the double refraction. It is therefore necessary to pass the light beam through the liquid exactly normal to the direction of propagation of the sound beam.

Test Cell and Transducer.—As a plane progressive sound wave is required, all the sound energy must be absorbed after passing through the liquid, and the acoustic intensity at the point of measurement must also be known.

With a transducer of the dimensions used the radial intensity distribution is far from uniform, but it was found that by introducing a mask with a suitable aperture a well defined beam can be obtained with a uniform intensity distribution.

There are several effects which set an upper limit to the acoustic intensities which can be used. They are: (*a*) Cavitation, which is the formation of small gas bubbles that diffuse the sound wave. (*b*) Bending of the light beam by the regions of the varying refractive index set up by the passage of the ultrasonic wave. (*c*) The temperature rise produced by the absorption of the sound waves. This was reduced to a minimum by suitable temperature stabilization and removing the chief sources of heat (the transducer and the absorber) as far away from the point of measurement as possible. By working with intensities of less than 0·4 watts/cm.^{2}, cavitation and diffraction effects could be neglected.

Description of the Apparatus

The apparatus consists of a transducer which radiates an acoustic wave into a test cell and an acoustic absorber which is attached to the bottom of the cell. This arrangement is completely immersed in a constant temperature bath. The general set up is shown in Fig. 1. A beam of light from a high pressure mercury arc passes through the cell after being collimated and polarized. The light emerging from the cell then passes through a diaphragm, the Wollaston prism, the Faraday coil, the analyzer and finally into an observation telescope.

In considering the apparatus in detail, the optical system, the test cell and the transducer will be treated separately.

The Optical System

This is shown diagrammatically in Fig. 1. It consists of two optical benches 0·75 and 0·5 metres long bolted to a 6 ft. iron girder, with the test cell placed at the centre. The light source S is a 90 W Phillips high pressure mercury arc. The filter F_2 is a Wratten 77A, which is protected from the heat of the lamp by Chance ON 19 heat resisting glass F_1. (95 per cent light transmission and 5 per cent heat transmission). The lens L_1 produces an image of the arc at D_1, and lens L_2 is set to give a parallel beam of light. Diaphragm D_2 limits the width of the beam passing through the polarizer to a diameter of 3 mm. The polarizer and analyzer are identical Glan Thompson prisms mounted on divided circles which can be read to 2′ of arc. The vibration direction of the polarizer is set at 45° to the vertical. After leaving the cell, the light beam passes through a diaphragm D_3 which is about 2 mm. diameter. This ensures that a well-defined image of uniform brightness is seen through the observing telescope which is focused on D_3.

The Faraday coil employs a lead-zinc borate glass (Chance DEDF No. 030,181, Verdet constant about 0·14 min/Gilbert.) in the form of a cylindrical rod 5 cm. long 1 cm. diameter. The glass is surrounded by a solenoid 8 cm. long consisting of 38,000 turns of 42 S.W.G. wire. A current of 6 mA through the coil

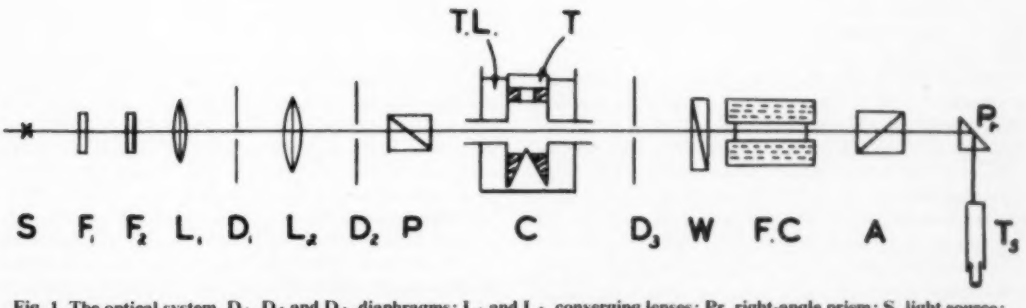

Fig. 1. The optical system. D_1, D_2 and D_3, diaphragms; L_1 and L_2, converging lenses; Pr, right-angle prism; S, light source; F_1, heat filter; F_2, Wratten 77A mercury green filter; P, polarizer; A, analyzer; W, Wollaston prism; Ts, telescope; F.C., Faraday coil; C, cell; T, transducer; T.L., thermostat liquid.

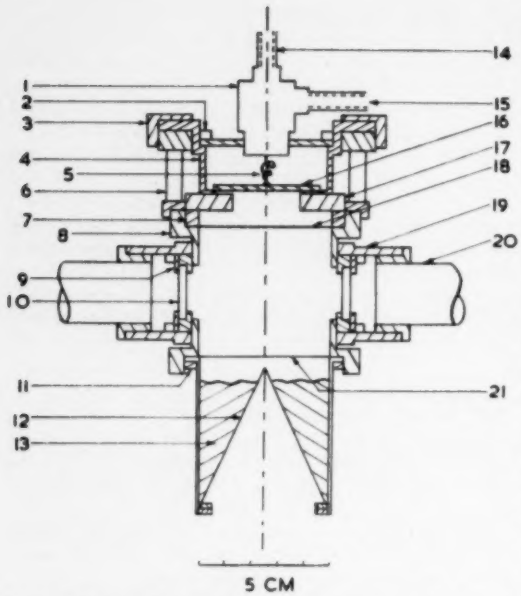

Fig. 2. The test cell and transducer. 1, R.F., plug; 2, 3, 7 and 11, retaining rings; 4, transducer housing; 5, current lead; 6, spacer; 8, cell; 9 and 10, window assembly and window; 12, reflecting cone; 13, sound absorber; 14, co-axial line to matching network; 15, co-axial line to V.V.M.; 16, ceramic disc; 17, mask; 18 and 21, ultrasonic windows; 19, window housing; 20, light tube.

produces a rotation of the order of 0·5°. The current is supplied through a potential divider arrangement from a 60v battery, and is measured by measuring the voltage drop across a standard resistor by means of a chart recorder. (10 mV full scale deflection).

The Transducer

The transducer consists of a lead zirconate (Brush LZ4A) ceramic disc 4 cm. diameter 1·9 mm. thick. The disc is silvered on both faces and cemented to the stainless steel housing using a Johnson Matthey thermo-setting silver preparation. The current lead of braided copper wire is attached to the top surface of the disc by means of this silver cement. The transducer is shown diagrammatically in Fig. 2.

The electric power required to drive the transducer is obtained from a conventional type of R.F. amplifier using a pair of 807 beam tetrode valves in push pull. The current is fed to the transducer through a 50 ohm. coaxial cable, which is terminated in an artificial line matching network to match the cable impedance to the low impedance (about 10 ohms.) of the transducer. The transducer is operated at 0·99 mc/s and 3·23 mc/s, and provision has been made to work at higher modes when required.

The mask, which is made of absorbing material, has an aperture of 2·5 cm. diameter, which gives a well defined beam of about 2·0 cm. diameter with a uniform radial intensity distribution. Using this arrangement 40 per cent of the total electrical energy applied to the transducer is transformed into useful acoustic energy.

The Test Cell

The test cell (Fig. 2) is a brass cylindrical container 5 cm. diameter with a capacity of approximately 100 cc. The wall thickness was reduced to a minimum by milling the outside of the cylinder, and the window housings (19) attached to one pair of the parallel faces. The light beam enters and leaves the cell through brass tubes (20). The inside of the cell is platinum plated to facilitate cleaning, and the top and bottom consists of 0·001 in. thick polythene tape ((18) and (21)) which allows the passage of the ultrasonic waves without serious attenuation.

The ultrasonic absorber consists of a conical reflector (12) made of thin brass foil fitted into a cylinder. The space between the cone and the cylinder is packed with cotton wool (13), and then filled with the liquid under test. The absorber is attached to the cell by means of a retaining ring (11).

The cell is placed inside a thermostat tank. The thermostat liquid is water, and the circulation is so arranged that there is a continuous flow of water inside the cone, which ensures that the heat generated in the absorber is carried away. The thermostat box is lagged with glass fibre and placed on a turn table which can be adjusted in the vertical and horizontal planes.

When the thermostat liquid completely covers the cell the absorbing mask is placed in position. The transducer, which is fixed into a stand is then placed on top of the cell. In this way the acoustic wave is transmitted through the thermostat liquid into the cell, and most of the heat generated in the transducer is carried away.

Fig. 3 shows a photograph of the apparatus.

Precision of Measurements

The precision of measurement of which the apparatus is capable is governed by the precision by which (1) the birefringence and (2) the acoustic intensity can be measured. These two factors will be considered separately.

Birefringence Δn.—The induced birefringence is measured by increasing the current through the coil until the two images are of equal brightness. The double refraction is then related to the rotation by equation (2). When measuring a mean rotation of 7·8′ under ideal conditions of observation a maximum deviation from the mean of 0·6′ and a standard deviation of 0·4′ has been obtained which corresponds to a birefringence of the order of $1{\cdot}5 \times 10^{-9}$ or $1{\cdot}0 \times 10^{-9}$ respectively. This 'ideal' condition is governed to a great extent by the undeviated image produced by the Wollaston. The best conditions for observation were obtained when the intensity of the two deviated images was considerably greater than that of the undeviated image, but not too great so as to make the

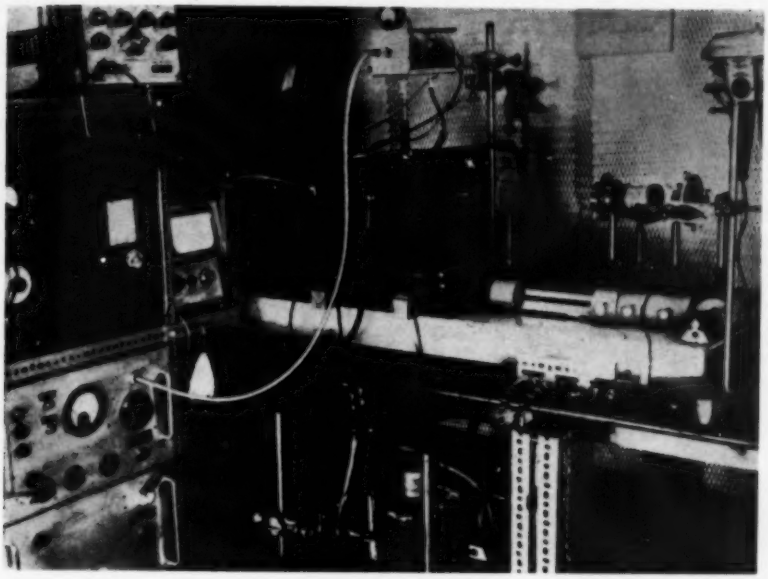

Fig. 3. Photograph of the apparatus showing the ultrasonic generator, thermostat box and optical system.

estimation of equal brightness difficult. This corresponded to rotations between four and 25 minutes.

The value of the rotation measured is governed by the calibration constant of the Faraday coil. This is determined by rotating the analyzer by a known amount and noting the current required to give equal brightness. The current required to produce a rotation of 1′ was of the order of 0·245 mA $\pm$ 0·5 per cent.

Acoustic Intensity $\bar{\omega}$.—The measurement of acoustic intensity to any degree of accuracy is very difficult. The method described by Willard (1949) is employed, in which a parallel beam of light from a slit source is diffracted by the progressive wave system. The intensity of the various diffraction orders varies according to the corresponding Bessel functions, and can be calculated in terms of the acoustic intensity. The zero, first and second order zero intensities are suitable reference points. The validity of these measurements is not certain (Braezeale and Heidemann, 1959) but the values obtained compare favourably with radiation pressure balance measurements. Using water as a test liquid the ratio of the acoustic power to the electrical power applied to the transducer has been determined. A mean value of 0·38 with a standard deviation of 0·045 was obtained.

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Forthcoming Conferences

Feigl Anniversary Symposium 1962

The Midlands Section of the Society for Analytical Chemistry is holding an International Symposium on Analytical Chemistry at the University, Edgbaston, Birmingham, 15, England, during the period April 9 to 13, 1962, inclusive. This is being held in honour of Prof. F. Feigl to commemorate his 70th birthday.

The programme will consist of:

(*a*) Contributions on original scientific work.
(*b*) Reviews of recent advances in selected branches of analytical chemistry.
(*c*) Exhibition of new scientific equipment.

Following the Symposium there will be a half-day Conference, on the morning of Friday, April 13, dealing with the 'Teaching of Analytical Chemistry'.

To this all delegates are invited.

The social programme will include a civic reception to the delegates and their ladies, a Symposium dinner, informal luncheons and dinners and a special programme arranged for lady visitors.

Plenary lectures will be given by Prof. R. Belcher (U.K.), Prof. Dr. Ing. F. Feigl (Brazil) and Prof. P. W. West (U.S.A.).

Application forms and further information can be obtained from: Honorary Symposium Secretary, M. L. Richardson, A.R.I.C., A.C.T.(Birm), c/o John & E. Sturge Ltd., Lifford Chemical Works, Lifford Lane, Kings Norton, Birmingham, 30.

THE STATIC MEASUREMENT OF YIELD STRESS

by G. Boardman,* B.Sc., M.Sc., and R. L. Whitmore, B.Sc., Ph.D., D.Sc., F.Inst.P.

University of Nottingham

Introduction

The yield stress of a fluid possessing time-independent rheological properties is usually obtained by extrapolating the flow-curve of shear-stress against shear-rate, obtained for the fluid from viscometer measurements, to zero shear-rate. This procedure cannot in general be expected to give an accurate value of the yield stress for one of two reasons:

(*a*) If the observed flow-curve covers only a region of comparatively high shear-rates there may be curvature in the unobserved low shear-rate region, making extrapolation unreliable.

(*b*) If measurements are made down to very low shear-rates, plug-flow and slippage may affect the readings obtained and appropriate corrections can only be applied when the flow characteristics of the fluid are already known.

To obtain a more reliable value of yield stress a measurement which is made under virtually static conditions is required. If a body is at rest in a fluid possessing a yield stress and a force is applied to it, the body will not move until the applied force exceeds the force exerted by the yield stress of the fluid on the body. Measurements of this force on suitable bodies constitute the technique described in this paper. A similar technique has been described previously by McMillen (1932) and Evans and Reid (1936) but in neither case were the limitations examined nor the effect of the various variables on the results obtained investigated.

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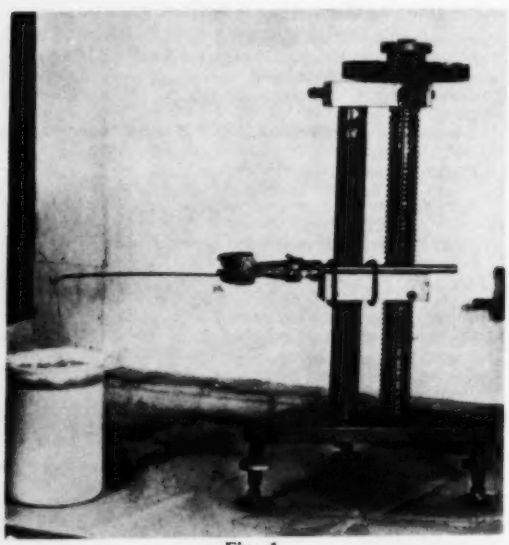

Fig. 1.

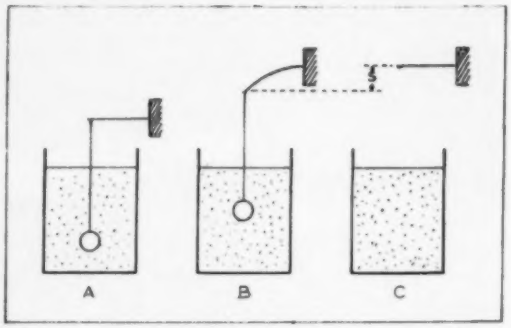

Fig. 2. Principle of operation.

Principles

The apparatus, which is shown in Fig. 1, consists of a rigid stand carrying a bracket which can be moved vertically on a screw thread. One end of a horizontal wire is fixed rigidly to the bracket and the other is bent into a hook.

The principle of the method is shown in Fig. 2. In position A a body is suspended in the fluid from the hook of the horizontal wire by means of a thin thread. The bracket holding the fixed end of the wire is then slowly raised to position B. In the absence of a wall layer, the effect of which will be considered later, the free end of the wire is at first stationary because the immersed body is held fast by the yield stress, so that the wire bends and an elastic restoring force is set up in it which is transmitted to the body. When the body commences to move through the fluid the maximum force which the yield stress can exert acts downwards on the body and, together with the body's apparent weight, is in equilibrium with the restoring force in the wire which is given by the sag of the free end of the wire.

The height of the free end of the wire in position B is measured with a travelling microscope and the wire is then unloaded, giving position C. The new height of the free end is measured and the change in height, s, found.

Then $s = c(F_s + W')$ ..... (1)

where F_s = maximum force exerted by the yield stress.

W' = apparent weight of the body in the fluid.

c = calibration constant of the wire.

In order to obtain a second set of readings, the bracket is lowered to a position approximating to A and the lifting procedure repeated.

Before F_s can be calculated the apparent weight W′ of the body in the fluid must be known. For a Newtonian fluid of density ρ, it follows from Archimedes' principle that the apparent weight W′ of an immersed body is given by

$$W' = W - \rho V g \quad \ldots\ldots\ldots (2)$$

where V and W are the volume and true weight of the body respectively.

This relationship cannot be assumed to hold for fluids possessing a yield stress because they behave partly as solids and do not necessarily transmit pressures hydrostatically, as required by Archimedes' principle.

The applicability of Archimedes' principle to aqueous suspensions of flocculated China Clay was tested on a 20 per cent suspension (by weight) by measuring s for a set of bodies having equal areas but different weights. The set was made by sticking together, face to face, varying numbers of identical, thin, metal laminae. They were suspended from the calibrated wire with their flat faces horizontal and their thickness was sufficiently small to be neglected.

On plotting $\frac{s}{c}$ against the number of laminae forming the body, n, Fig. 3 was obtained. If Archimedes' principle holds, the apparent weight of each body is, from equation (2)

$$W' = nw - n\,vg = n\,w' \quad \ldots\ldots (3)$$

where v, w and w′ are the volume, true weight and Archimedian apparent weight respectively of a single lamina. Equation (3) gives a value of $w' = 2{,}586 \pm 10$ dyne for the bodies used.

By equation (1) Fig. 3 should be a straight line of equation

$$\frac{s}{c} = F_s + w'\,n \quad \ldots\ldots\ldots (4)$$

Its linearity is confirmed and the slope of the line gives

$$w' = 2{,}629 \pm 40 \text{ dyne}$$

Thus Archimedes' principle appears to hold to within about 2½ per cent for clay suspensions, this range of variation being insignificant compared with other errors in the method.

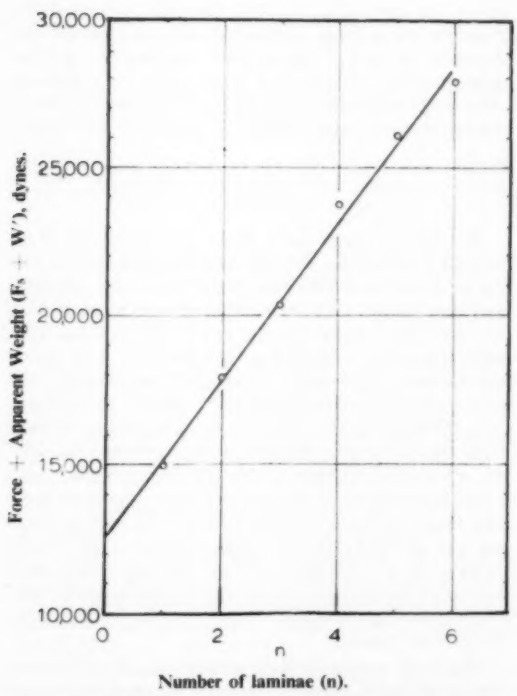

Fig. 3. Applicability of Archimedes' Principle.

When the wire is brought into position B some upward creep of the free end occurs. If the yield stress is defined as the least stress required to cause two adjacent laminae to move past one another, the replacement of one of the laminae by a solid plate is likely to produce a different value of the yield stress because the linkages will be different. A layer of modified fluid (of thickness d) adjacent to the surface can be assumed to be present (Reiner, 1960) and to possess Newtonian flow-properties, its fluidity being ϕ_w. It is also assumed that the bulk fluid satisfies the Bingham equation

$$S = S_0 + \frac{D}{\phi_B}$$

where S = shear stress exerted on the fluid, S_0 = yield stress of fluid, D = rate of shear of fluid, and ϕ_B = fluidity of the fluid.

Let v be the constant, upward velocity of the fixed end of the wire when moving from position A to position B, and V be the instantaneous, upward velocity of the free end.

The additional stress, S, acting on the lamina, in excess of that needed to balance its apparent weight is given by:

$$\frac{dS}{dt} = \frac{c}{A}(v - V), \text{ while the wire is rising} \quad \ldots\ldots (5)$$

$$\frac{dS}{dt} = -\frac{c}{A}V \text{ after the raising has ceased} \quad \ldots\ldots (6)$$

where A is the total surface area of the lamina.

When $S > S_O$

$$V = [\phi_w d + \phi_B (x - d)]\,S - \phi_B (x - d)\,S_O \quad \ldots (7)$$

where x = width of Bingham fluid which shears when the yield stress is exceeded.

When $S < S_O \quad V = \varnothing_W \, dS$ .... (8)

On substituting for V in equation (6) from equations (7) and (8), and integrating,
when the wire is stopped in position B,

(*a*) $S > S_O$, for a time t_1, $S =$

$$\frac{\varnothing_B (x - d) S_O}{\varnothing_W d + \varnothing_B (x - d)} + \left[S_{max} - \frac{\varnothing_B (x - d) S_O}{\varnothing_W d + \varnothing_B (x - d)} \right]$$

$$\exp \left(- \frac{c t_1}{A} \left[\varnothing_W d + \varnothing_B (x - d) \right] \right) \quad \ldots (9)$$

Where S_{max} = maximum shear stress attained

(*b*) $S < S_O$, after time t_1,

$$S = S_O \exp \left[- \frac{c}{A} \varnothing_W d \, (t - t_1) \right] \quad \ldots (10)$$

The exponential decay of equation (10) represents the creep and by making measurements of the variation of S with t for the clay suspensions examined, the value of $\varnothing_W d$ was found to be $3{\cdot}2 \times 10^{-6}$ c.g.s. The existence of such a low value of $\varnothing_W d$ indicates (from equation (8)) that S_O is exceeded very early in the raising process and the substitution of all reasonable values of x in equation (9) shows that S falls to S_O within a few seconds of position B being reached. If the deformation of the wire is measured some 30 seconds after position B is reached the resultant yield stress is likely to be 1-2 per cent below the true value.

Practice

(*a*) *The Suspending System.*—For the clay suspensions examined, a hard brass wire 17 cm. in length and 0·15 cm. in diameter was found satisfactory. It was calibrated by adding known weights to its tip and measuring the deflections produced. The distance from position A to position B was approximately 4 cm. and the speed of raising was always low enough to prevent vibrations being transmitted to the free end.

The bodies were suspended by a very thin, smooth wire on which yield stress effects in the fluid could be neglected. It was attached to the body by small pieces of waterproof adhesive tape in the minimum possible number of places. The fastenings were made on vertical or underneath surfaces in order to prevent the tape being pulled off the surface.

(*b*) *The Fluid.*—No special temperature control was used as many workers have found that yield stress is independent of temperature in the normal range of room temperatures (e.g. Bingham, 1922). Nothing was found in the present work to challenge this view.

Measurements on vertical laminae were independent of the position of the boundaries of the fluid but with other orientations and shapes of bodies it was necessary to ensure that in position B the walls were further away than one half the horizontal dimension of the lamina or body.

Several values were usually obtained for each orientation of each body used, the suspension being well stirred between readings.

(*c*) *The Suspended Bodies.*—(i) *Properties.*—The densities of the bodies were, whenever possible, made sufficiently great for them to sink in the fluid. Bodies possessing a small weight and a large surface area, however, had to be pushed down into position A with a glass rod.

The weights and surface areas of the large bodies were measured individually. All laminae were cut from the same thickness of metal and calibrated for surface area in terms of true weight by measuring the areas of a few laminae and constructing a graph of surface area against weight. The volume per unit surface area of the laminae, which was required for calculating their apparent weight, was found by weighing in water a group of them of known total surface area.

(ii) *Orientation.* Experiments with laminae indicated the importance of orientation on the calculated yield stress. The apparent yield stress acting on horizontal discs was from five to seven times greater than that acting on vertical discs, this ratio being remarkably constant for a large number of suspensions. It was shown in Section 2 that vertical laminae give a close approximation to the true yield stress and a mean value was obtained by using vertical laminae of various shapes and area and finding an averaged yield stress

$$= \frac{F_S}{A}.$$

(iii) *Shape.* The yield stress as calculated from vertical laminae was substantially independent of their shape. The value obtained from horizontal laminae, however, varied with the selected shape, but not with the size. Thus a disc gave a value about 25 per cent higher than a long thin rectangle. For rectangular blocks with their faces horizontal and vertical, the effects of true yield stress acting on the vertical surfaces, and effective yield stress on the horizontal surfaces were superimposed (Boardman and Whitmore, 1961) the mean value depending upon the shape of the block.

For a sphere, the total force exerted by the yield stress was about the same as for a horizontal lamina possessing the same horizontal cross-sectional area as the sphere. Thus a sphere gave an apparent yield stress 1·5 to 2·5 times as great as a vertical lamina of the same total area but roughly equal to the extrapolated flow-curve value.

The high apparent yield stresses found with some bodies probably resulted from an unusual shear and flow pattern in the fluid. This was examined by visual-

ization experiments and is described elsewhere. (Boardman, 1961).

(iv) *Surface*. Measurements were made on laminae possessing different types of surface finish but the resulting yield stress was not noticeably influenced by them, as is to be expected from the theory given above.

Typical Results

Results are reported for a suspension of Supreme Clay supplied by English Clays Lovering Pochin & Co. Ltd. The concentration was approximately 15 per cent by weight, and the density of the suspension 1·094 gm. cm.$^{-3}$.

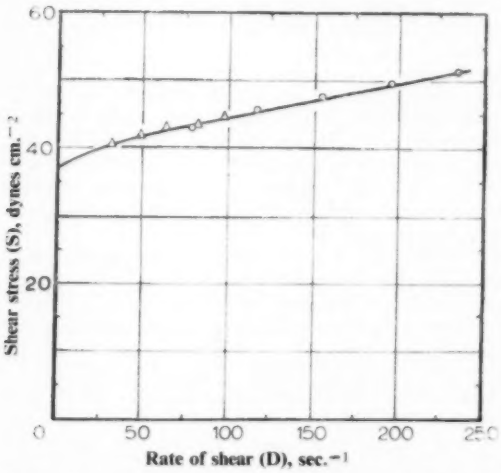

Fig. 4. Flow curve for Supreme Clay. Viscometer: Ferranti, V.H.A. Cylinders, 5 g. spring. ○ A.C. frequency 50 c/s.; △ A.C. frequency 21 c/s.

(*a*) *Flow Curve*.—This was obtained with a Ferranti co-axial rotating-cylinder viscometer. (Fig. 4). Extrapolation to zero rate of shear gives a yield stress of 38 ± 2 dyne cm.$^{-2}$.

(*b*) *Spheres*. (9 sizes from 0·5 cm. to 2 cm. diameter). Based on the surface area of the sphere, the yield stress equals 36·6 ± 2·5 dyne cm.$^{-2}$.

(*c*) *Horizontal Discs*. (Five diameters from 1 cm. to 3·5 cm.). Based on total surface area of each disc, yield stress equals 78·9 ± 5 dyne cm.$^{-2}$.

(*d*) *Vertical Laminae*. (Six different sizes; discs and rectangles).

(i) Smooth metal surface. Based on total surface area of each lamina, yield stress equals 16·0 ± 1 dyne cm.$^{-2}$.

(ii) Metal surface covered with rough adhesive tape. Based on total surface area of each lamina, yield stress equals 16·0 ± 1 dyne cm.$^{-2}$.

Thus the sphere and the flow curve gave a yield stress slightly more than twice the true value. The horizontal laminae gave a value almost five times too large.

ACKNOWLEDGMENT.—*The work was carried out as part of a programme sponsored by the National Coal Board to whom acknowledgment is made for financial assistance.*

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CORRESPONDENCE

To the Editor,

Laboratory Practice

DEAR SIR,—May I draw the attention of your readers to the advantages of joining the Food Group of the Society of Chemical Industry?

There is a belief in some quarters that it is necessary to have a scientific qualification to join the Society, but this is not so. Anyone having a serious interest in the technical side of the food industry will be welcomed into membership. While the primary interests of the Food Group are chemical, considerable attention is given to the technological, economic and legal aspects. The subscription to the Society is quite modest, and membership of all groups, with the exception of the Chemical Engineering Group, is free.

The Food Group is the largest, and holds meetings regularly from September to June, mostly in London but also elsewhere. Subjects for this year's programme include cereals, blackcurrant juice, meat flavour, low salt foods, apples, sugar, microbiological control, low carbohydrate foods, pork, gastronomy, foods low in gluten and in phenylalanine, and food technology in other countries. A special feature of the Group is the annual foreign tour when we visit factories in various European countries. This tour and the annual conversazione afford opportunities for members to get to know each other personally.

In addition to the Food Group, the Agriculture, Microbiology, Industrial Water, Chemical Engineering and other groups frequently hold meetings of interest to food technologists.

Information about the Society and application forms can be obtained from the Secretary, Society of Chemical Industry, 14 Belgrave Square, London, S.W.1. I shall be pleased personally to give assistance to anyone wishing to join the Society.

Yours faithfully,

J. G. DAVIS,

Chairman, S.C.I. Food Group.

9 Gerrard Street,

London, W.1.

ELECTRICAL METHODS FOR THE VISCOELASTIC BEHAVIOUR OF LIQUIDS UNDER CYCLIC SHEARING STRESS

by A. J. Barlow, G. Harrison, J. Richter, H. Seguin and J. Lamb

Department of Electrical Engineering, The University, Glasgow, W.2

Summary

Purely mechanical methods of evaluating the shear response of liquids are limited to comparatively low frequencies of cyclic stress of the order of a few hundreds of cycles per second. Before the introduction by Mason (1947) of a piezoelectric transducer as a means of viscosity measurement, investigations were confined to frequencies below about 20 kc/s. for which mechanical and electromagnetic devices were employed. In this range measurements on liquids having viscosities of a few poise or less showed no reduction of the dynamic viscosity with frequency, and elastic effects were absent.

The frequency range can be extended to about 200 kc/s. by the use of piezoelectric crystals vibrating torsionally. Measurements at higher frequencies, up to 10^8 c/s. or above, can be made using quartz crystals resonant in the thickness-shear mode. For still higher frequencies it is possible to generate shear waves by direct excitation of a quartz rod or disc inserted into a cavity resonator. Measurements of the shear modulus have been made at 1,500 Mc/s. and preliminary experiments at 3,000 and 10,000 Mc/s. are in progress.

In practice measurements are made of the components of the shear impedance, from which the associated values of dynamic viscosity and shear modulus are readily deduced.

A description is given of the following experimental systems:

1. *Resonant torsional quartz crystals operating in the frequency range 20 kc/s. to 150 kc/s. which are used for measurements of non-conducting liquids having viscosities below about 5 poise.*
2. *Torsional wave propagation in long rods immersed in a liquid. This method is applicable to liquids having viscosities above 5 poise and can be used over the frequency range 10 kc/s. to 100 kc/s.*
3. *Shear wave propagation in fused quartz and the measurement of the reflection coefficient at the interface between fused quartz and the liquid. Thickness-shear vibrating quartz crystals are employed as transducers over the frequency range 3-250 Mc/s.*
4. *Shear wave propagation in a rod of crystal quartz and the measurement of the reflection coefficient at a quartz-liquid interface. The crystal is directly excited by placing one end in a tunable cavity which can be made resonant in the frequency range 250 Mc/s. to 1,500 Mc/s.*
5. *Shear wave propagation through a thin quartz disc which is contained in a re-entrant cavity resonating at frequencies in the region of 3,000 Mc/s. The plane surfaces of the quartz disc are loaded by a thin film of the liquid under test.*

A description is given of the application of these various techniques to the measurement of the viscoelastic properties of liquids under high hydrostatic pressure.

Introduction

The measurement of viscoelastic properties of liquids under conditions of alternating stress is of practical importance in its application to problems of lubrication, particularly at high speeds. It is also of fundamental interest in the study of the liquid state, and the dependence of the flow of molecules on their composition and environment.

It is known that in some liquids the effective viscosity decreases with increasing rate of shear, but owing to the problems of excessive heat dissipation, it is difficult to perform reliable experiments at high rates of shear approaching 10^6 sec.$^{-1}$. Moreover, the lubrication engineer is interested in knowing the behaviour at these rates of shear and simultaneously under conditions of high hydrostatic pressures of several thousands of atmospheres, as are encountered in the lubricating film separating gear teeth. On the other hand, it is also known that liquids which exhibit such behaviour show a decrease of viscosity with increasing frequency of cyclic shear, and an associated increase in the modulus of rigidity, that is, of solid-like behaviour. These effects are amplitude independent under normal experimental conditions, and as the alternating stress measurements can be performed in a pressure vessel, a correlation is sought between this viscoelastic behaviour as a function of rate of shear in steady rotation and as a function of applied frequency in the alternating experiments.

It is now possible to work with cyclic stress waves varying from very low frequencies to thousands of megacycles per second and many of the interesting viscoelastic effects are to be observed at relatively high frequencies at atmospheric pressure. Measuring techniques covering the audio frequency range are well established (Markovitz *et al.*, 1952; Sittel *et al.*, 1954; Smith *et al.*, 1949). An outline is given here of methods which have been developed in our laboratory for use in the range of frequency from about 20 kc/s. to 3,000 Mc/s. Oscillating quartz crystals are used throughout; these techniques stem from the early work of Mason (1947), Mason, Baker, McSkimin and Heiss (1949) and McSkimin (1952).

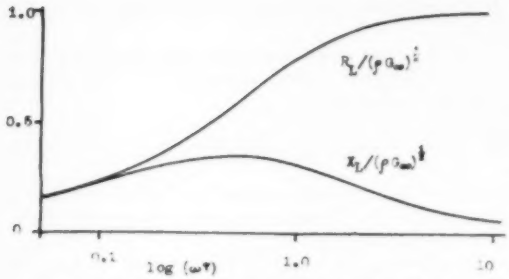

Fig. 1(*a*). Variation of the normalized shear impedance with frequency for a Maxwell fluid (single relaxation time).

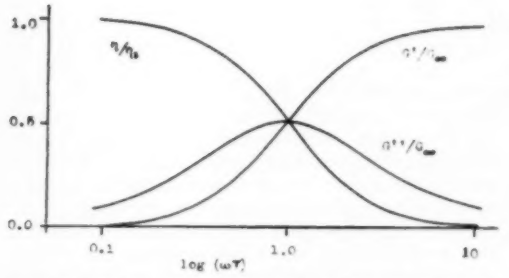

Fig. 1(*b*). Variation of the normalized shear modulus and dynamic viscosity for a single relaxation process.

Different experimental systems are employed over various ranges of the frequency spectrum, and a description is given of each, in turn, with some indication of the type of information to be found, although in the present context experimental results are used only to illustrate the measuring techniques.

The quantities which are obtained directly from experiment are the resistive and reactive components of the shear mechanical impedance ($Z_L = R_L + jX_L$) defined as the ratio of the shear stress to the rate of change of displacement transverse to the direction of propagation of the wave. These are related to the components of the complex shear modulus $G^* = G' + jG''$, defined as the ratio of shear stress to shear strain, by

$Z_L{}^2 = (R_L + jX_L)^2 = \rho(G' + jG'')$, where ρ is the density

Hence $G' = \dfrac{R_L{}^2 - X_L{}^2}{\rho}$, and $G'' = \dfrac{2R_L.X_L}{\rho}$

G' is the shear rigidity modulus which is zero at low frequencies ($R_L = X_L$, for Newtonian behaviour); and the dynamic viscosity, $\eta = G''/\omega$.

The liquid behaves elastically at very high frequencies and the dependence of the above parameters on frequency for a Maxwell fluid with a single relaxation time, $\tau = \eta_S/G_\infty$, is shown in Fig. 1, where η_S is the static viscosity and G_∞ the limiting shear modulus at high frequencies. In practice there is generally found a wide distribution of relaxation times which can be represented in terms of a spectrum.

Experimental Systems based on the use of Torsionally Vibrating Quartz Crystals: 20 kc/s. to 200 kc/s.

(a) *Resonant Torsional Crystals.*—Torsional vibration of a quartz crystal may be produced by cutting a cylinder so that the X-axis lies parallel to the length, and exciting it by means of four cross-connected electrodes placed as shown in Fig. 2. The electric fields in the Y-direction on either side of the line YY′ are of opposite sign and produce two opposing shear stresses. The cylinder reacts to these with a torsional motion and a slight lengthwise motion. The torsional motion is favoured by a large length to diameter ratio, and the maximum efficiency is obtained by operating the transducer at the frequency of a resonant mode. In the fundamental mode of oscillation the length of the transducer is equal to half a wavelength, and the frequency of this resonance is given by the expression

$$f = \frac{1}{2h(\rho Q\tau)^{\frac{1}{2}}} \text{ c/s} \qquad (1)$$

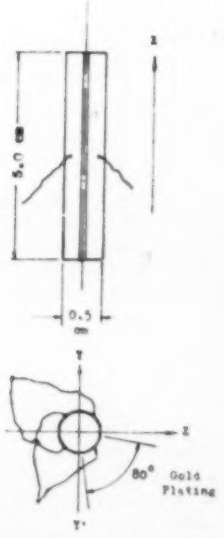

Fig. 2. Electrode arrangement for torsionally vibrating quartz crystal resonant at 38.6 kc/s.

where h is the length of the crystal, ρ_Q is the density of quartz and σ is the elastic modulus for torsion given by

$$\sigma = \frac{S_{55} + S_{66}}{2} = 2{\cdot}42 \times 10^{-12}\ \text{cm.}^2/\text{dyne} \qquad (2)$$

In practice quartz cylinders can be manufactured with lengths between about 1 and 10 cm. corresponding to resonant frequencies of 2×10^5 c/s. and 2×10^4 c/s., respectively.

The vibration of the crystal is damped by immersing it in a liquid to be measured and the properties of the libuid in shear may be calculated from the resulting changes in the resonant frequency and the resistance at resonance compared with the corresponding values in vacuum. Denoting the resonant frequency and the resistance of the crystal in vacuum by f_O and R_O, the real (R_L) and imaginary (X_L) parts of the shear impedance, Z_L, of the liquid are related to the resonant frequency and resistance of the crystal in the liquid f_R and R_R by the following equations:

$$f_R = f_O - K_2 . X_L \qquad (3)$$

$$R_R = R_O + K_1 . R_L \qquad (4)$$

K_1 and K_2 are constants for a particular crystal under given conditions of temperature and pressure and may be determined either by calculation from the crystal dimensions, or from experiments with liquids of known shear impedance. These constants are related to the length h, radius r and density ρ_Q of the crystal as follows:

$$K_1 = \frac{4L}{\rho_Q}\left\{\frac{1}{r} + \frac{1}{h}\right\} \qquad (5)$$

$$K_2 = \frac{1}{\pi\rho_Q}\left\{\frac{1}{r} + \frac{1}{h}\right\} \qquad (6)$$

The quantity L is the effective electrical inductance of the crystal in vacuum: the inductance of the crystal in a liquid L′ may be determined from the difference in the frequencies f_1 and f_2 at which the resistance is twice the value at resonance (R_R) measured in any liquid, using the equation

$$L' = \frac{R_R}{2\pi(f_2 - f_1)} \qquad (7)$$

Only slight changes in inductance of the crystal are caused by immersing it in a liquid, and the value pertaining to the crystal in vacuum is given by

$$L = L'\left\{1 - \frac{2(f_O - f_R)}{f_R}\right\} \qquad (8)$$

This correction is generally small and less than 1 per cent.

For a liquid exhibiting Newtonian behaviour, the shear impedance is related to the density ρ and viscosity η by the expression

$$Z_L = R_L + jX_L = (1 + j)\,[\pi f \eta \rho]^{\frac{1}{2}} \qquad (9)$$

Therefore the real and imaginary parts of the impedance are equal in magnitude: use is made of this property in determining the constant K_2.

The value of the constant K_1 can be determined in two ways:

(*a*) from measurements of a Newtonian liquid of known R_L obtained from measurements of density and viscosity of the liquid in question, then

$$K_1 = \frac{R_R - R_O}{[\pi f \eta \rho]^{\frac{1}{2}}} \qquad (10)$$

(*b*) by measuring the value of the inductance, L, using equations (7) and (8) with any liquid and substituting this in equation (5). This method has the considerable advantage that the density and viscosity of the liquid need not be known. These two methods give the same value for K_1 to within the experimental error of ± 1 per cent. Variation of K_1 with temperature and with pressure is shown in Fig. 3 and 4 respectively.

The constant K_2 can only be determined accurately from measurements of liquids exhibiting Newtonian behaviour, when $X_L = R_L$, R_L being determined either from a knowledge of K_1, or from measurements of the density and viscosity of the liquid. Then K_2 is given by

$$K_2 = \frac{f_O - f_R}{X_L} \qquad (11)$$

Values of K_2 given from the crystal dimensions using equations (6) are in general 5 per cent below the values obtained by the above experimental method. This difference is probably due to the effect of the electrodes and mounting wires, and in practice the values of K_2 have been obtained using equation (11), with a Newtonian liquid ($X_L = R_L$).

Initial experiments made on a crystal with a ground surface finish showed that K_2 was dependent upon the viscosity of the liquid in question. However, when this same crystal was polished and replated, K_2 was found to be constant with liquids above about 2 centistoke in viscosity. Deviations occurred in liquids of lower viscosity, but presumably these could be removed by a finer polishing procedure. This experiment showed conclusively that deviations of K_2 from a constant value for different liquids is a result of surface roughness on the crystal. This is reasonable in view of the fact that the attenuation of the viscous wave in the liquid is very high, the distance of propagation for the amplitude to

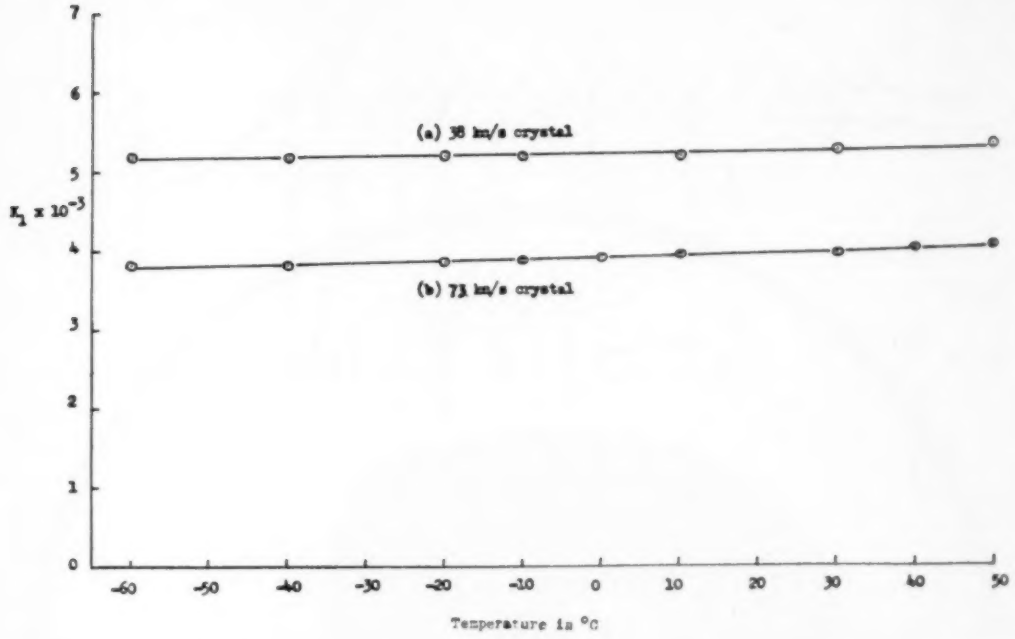

Fig. 3. Resonant torsional crystals: variation of constant K_1 with temperature. (*a*) 38 kc/s. resonant frequency crystal. (*b*) 73 kc/s. resonant frequency crystal.

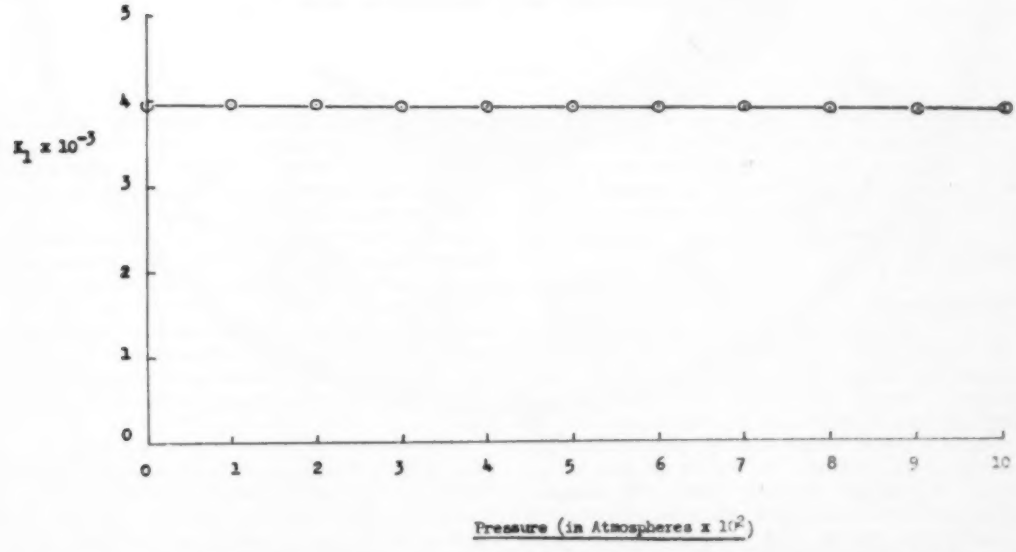

Fig. 4. Resonant torsional crystal: variation of constant K_1 with pressure for the 73 kc/s. resonant frequency crystal at 30°C.

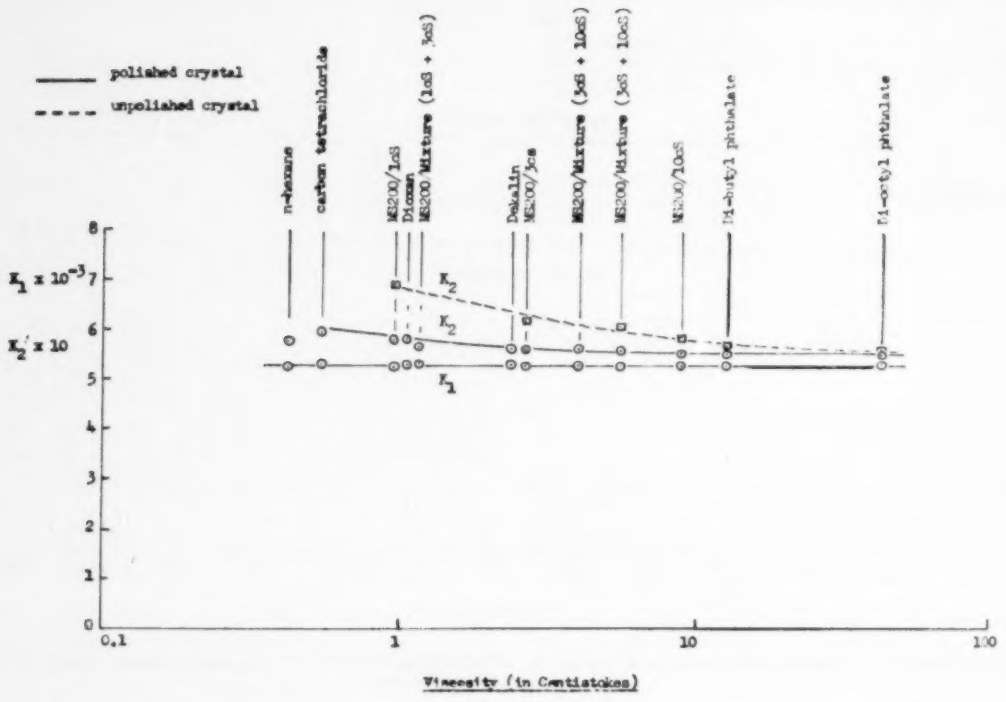

Fig. 5. Resonant torsional crystals. Measured values of K_1 and K_2 for liquids of different viscosity. The dashed curve is for the crystal with a ground surface and the full lines for the polished crystal.

be reduced to l/e of the initial value is only $1{\cdot}5 \times 10^{-4}$ cm. for a liquid of 1 centipoise viscosity at a frequency of 70 kc/s.: this distance is comparable with surface irregularities of the rough crystal. The constant K_1 was not found to vary with surface roughness of the crystal. The dependence of K_2 on temperature is shown in Fig. 6.

Electrical System

Fig. 7 is a schematic diagram of the measuring system. The crystal forms one branch of a resistance and capacitance bridge (Wayne-Kerr type B601 radio frequency bridge). The output from the bridge is amplified and displayed on an oscilloscope and the bridge is fed from a high stability, variable frequency oscillator, the frequency of which is measured by means of a digital frequency meter. The crystal is connected to the measuring terminals by two lengths of coaxial cable, and the outer braiding of these leads is connected to the centre tap of the bridge input transformer in order to eliminate the large shunt capacitance of the leads from the measurements. The capacitance of the cables is thus in shunt across the transformers and so does not affect the balance of the bridge. The impedance measured by the bridge is therefore that of the crystal only, the shunt impedance of the leads being eliminated.

The resonance curve of the crystal in vacuum has a width of about 1·5 c/s. and in order to make accurate measurements of the resonant frequency the oscillator must be stable to better than 0·1 c/s.; this represents a stability requirement of $\pm$ 1 in 10^6. The oscillator consists of a very stable master oscillator, operating in the range 1 to 2 Mc/s., and contained in a temperature controlled enclosure, followed by a series of bistable stages which divide the frequency down to the required value.

A digital frequency meter with an accuracy of $\pm$ 1 part in 10^6, $\pm$ 1 count, is used to measure the frequency of the oscillator. The oscillator is stable enough to allow a ten second counting time to be used so that the error in the frequency measurement does not exceed $\pm$ 0·2 c/s. for frequencies below 100 kc/s.

This technique can be used for measurement of the shear impedance of liquids over wide ranges of tem-

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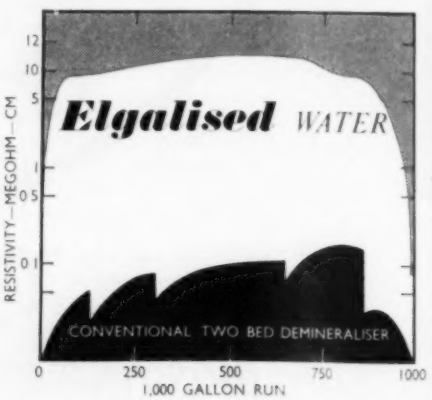

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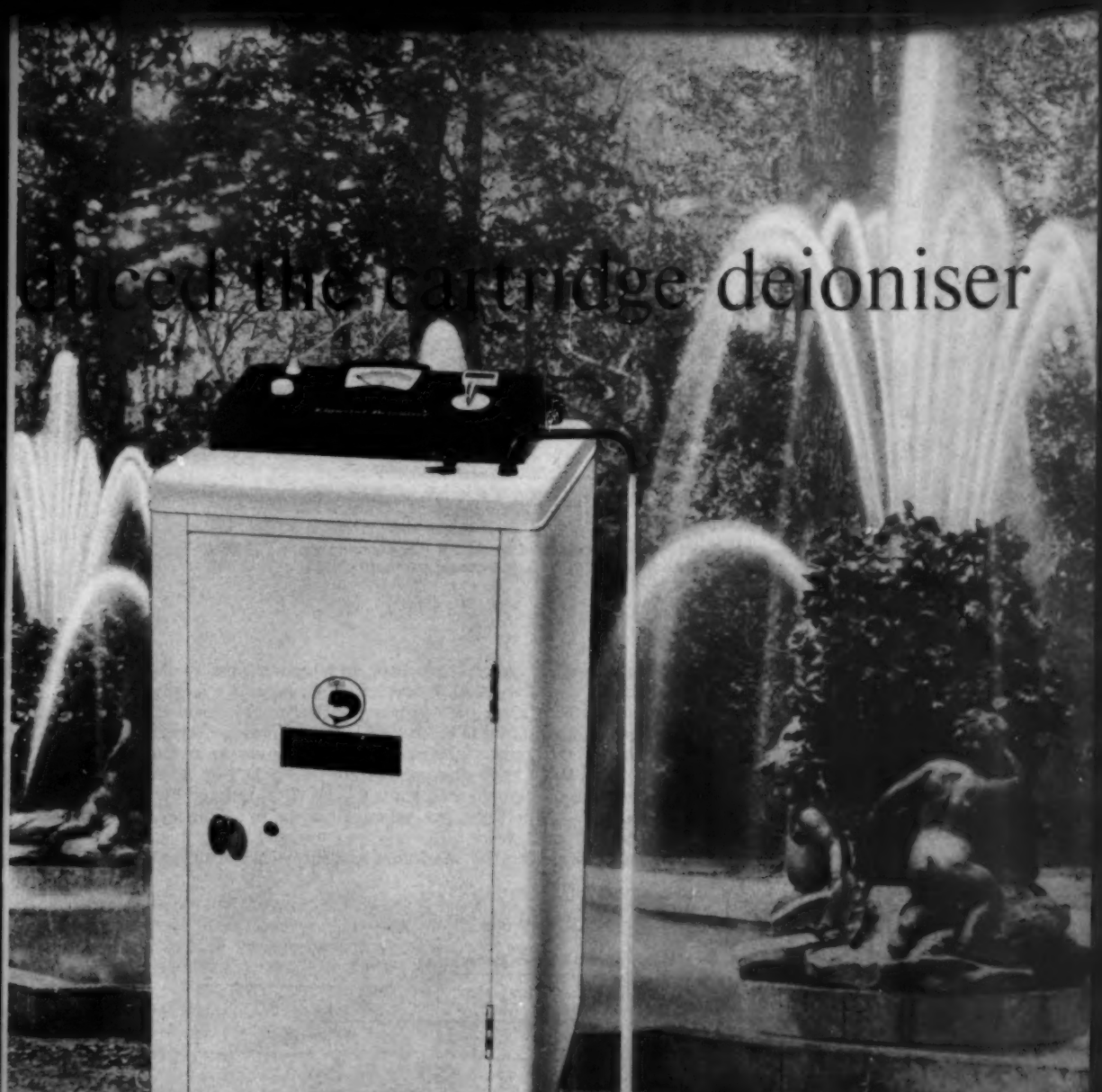

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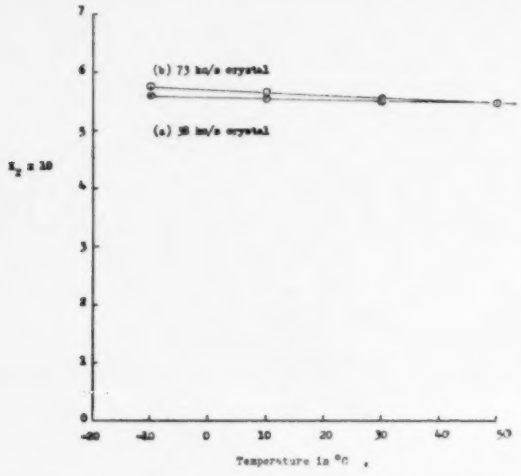

Fig. 6. Resonant torsional crystals: variation of constant K_2 with temperature. (*a*) 38 kc/s. resonant frequency crystal. (*b*) 73 kc/s. resonant frequency crystal.

perature. Results obtained using three different crystals are shown in Fig. 8 for the product of viscosity and density for several low viscosity liquids: these values agree with those obtained by conventional methods to within $\pm$ 2 per cent. Measurements made of the viscosity of toluene down a temperature of $-60°$ agree with values obtained by conventional methods to better than the estimated experimental accuracy of $\pm$ 2 per cent. These techniques enable the viscosity of liquids under high pressure to be determined with greater ease than with conventional methods. The practical difficulties of the latter are largely removed since the oscillatory technique only requires electrical leads to be brought out of the pressure vessel. Measurements obtained on several liquids up to pressures of 1,000 atmospheres are shown in Fig. 9, in which a comparison is made with data obtained by Bridgman (1926). The values all agree to within experimental error.

The maximum viscosity which can be measured

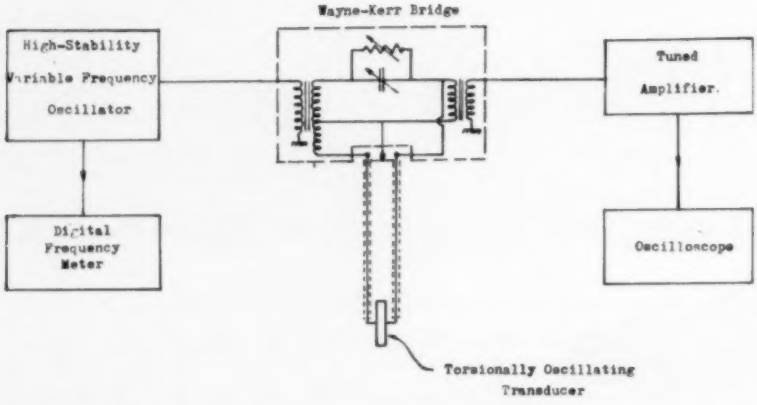

Fig. 7. Schematic diagram of the measuring system using the resonant torsional crystals.

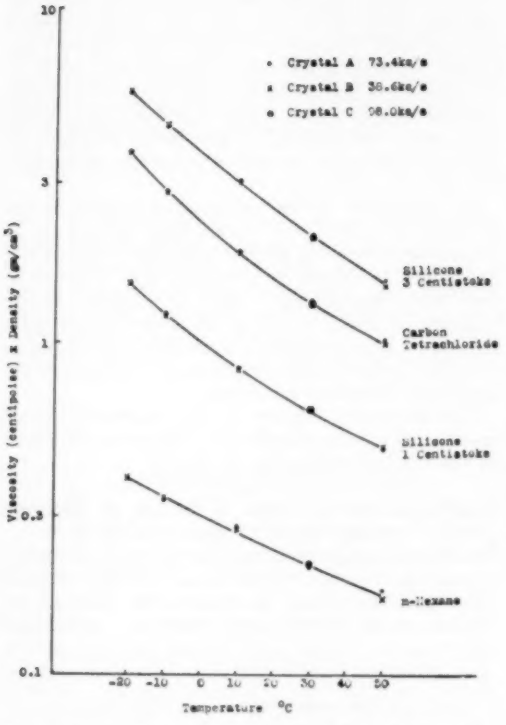

Fig. 8. Measured values of the product viscosity x density obtained using three different resonant torsional crystals. • crystal A, 73.4 kc/s.; x crystal B, 38.6 kc/s.; o crystal C, 98.0 kc/s.

using this technique is about 5 poise. With liquids of higher viscosity, the resistance at resonance is too high (over 10 MΩ) to be measured accurately, and a modification of this method is used as described below.

(b) *High Viscosity Technique: Travelling Wave Method.*—In this system a short pulse of torsional waves is propagated along a long thin rod. This pulse is produced by a torsional crystal rigidly fixed to the end of the rod. The pulse is propagated along the rod to the end, where it is reflected, and returns to the crystal where it generates a received pulse. It can be shown that the changes of amplitude (A) and phase (B) of this received pulse, which results from the immersion of a length h of the rod in a liquid, are related to the liquid impedance Z_C by the equation:

$$Z_C = \frac{\rho.v.r}{2h}(A + jB) \qquad (12)$$

where ρ is the density of the rod material, r is the radius of the rod, and v the velocity of propagation of torsional waves in the unloaded rod. $Z_C = R_C + jX_C$, is the cylindrical wave impedance. The plane wave impedance Z_L, can be obtained from this using the equation:

$$Z_L = \left\{ R_C - \frac{3}{\omega\rho_L r} . R_C.X_C \right\} + j\left\{ X_C + \frac{3}{\omega\rho_L r} \frac{(R_C^2 - X_C^2)}{2} \right\} \qquad (13)$$

Electrical System

Fig. 10 is a schematic diagram of the electrical system. The output of the high stability oscillator is gated at intervals of 1/25 second to provide pulses of about 0·5 millisecond duration and 100 volts peak to peak amplitude. These pulses are applied to the transducer. The received signal is taken through a buffer amplifier and added to an attenuated continuous wave from the oscillator. The resulting waveform is further amplified and displayed on an oscilloscope. By suitable adjust-

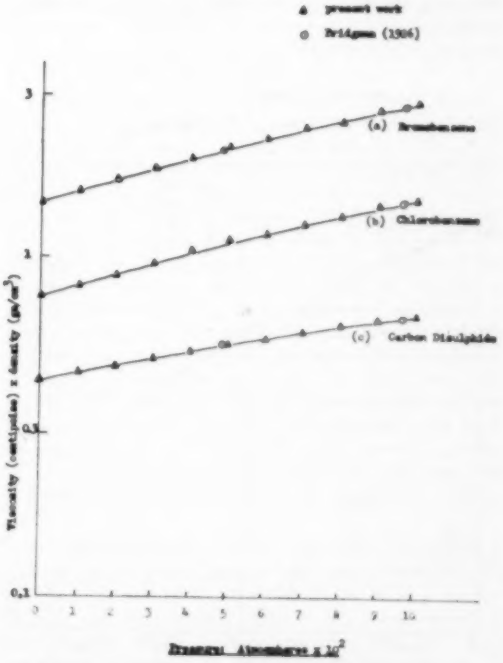

Fig. 9. Measured values of viscosity as a function of pressure obtained by using a torsional quartz crystal operating at its fundamental resonance frequency of 73.4 kc/s. Comparison is made with data obtained by Bridgman (1926). Curve (*a*) bromobenzene, curve (*b*) chlorobenzene and curve (*c*) carbon disulphide.

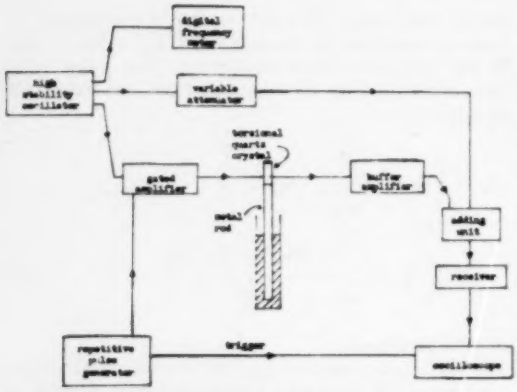

Fig. 10. Schematic diagram of the electrical system using travelling torsional waves for liquids having viscosities exceeding 5 poise.

ment of the attenuator and the oscillator frequency any received pulse may be exactly cancelled against the continuous signal. On surrounding the rod with a liquid, the changes in attenuator setting and oscillator frequency necessary to restore cancellation are determined.

This technique has been used for measurements in the frequency range 10 kc/s. to 100 kc/s., using torsional quartz crystals with resonant frequencies ranging from 20 kc/s. to 100 kc/s. attached to nickel silver rods 50 or 100 cm. long. Each crystal is fixed to a rod of the same diameter by a thin film of resin adhesive. Operation of the system is not confined to the resonant frequency of the crystal since the transducer is heavily damped by the rod.

The sensitivity of the technique is limited by the accuracy to which the cancellation point can be determined. With the present apparatus the possible errors are $\pm$ 0·1 db. in amplitude and $\pm$ 1 c/s. in frequency measurement. This corresponds to an accuracy of about $\pm$ 70 $\pm$ j40 ohms in the shear impedance measurements; the viscosity of a liquid of 10 poise viscosity can be determined to about $\pm$ 3 per cent in the absence of relaxation.

Typical results obtained by the resonant torsional crystal and the travelling wave techniques are shown as 'universal curves' in Fig. 11 and 12.

Experimental System Based on the Use of Thickness-Shear Vibrating Quartz Crystals: 3 to 250 Mc/s.

The method employed to measure the shear mechanical impedance of liquids between 3 and 100 Mc/s. is based upon the measurement of the complex reflection coefficient of an incident shear wave at a solid/liquid

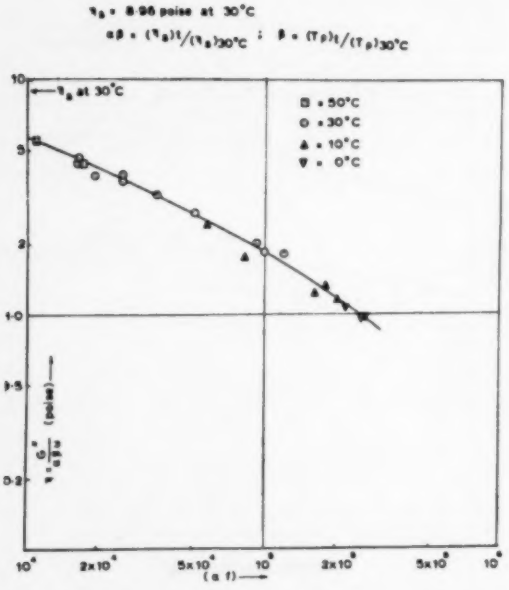

Fig. 11. Plot of dynamic viscosity versus (αf) for MS 200/1000 cS dimethyl silicone normalized to 30°C. η_s = 8.96 poise at 30°C.; $\alpha\beta = (\eta_s)_t/(\eta_s)_{30°C.}$; $\beta = \frac{(T\rho)_t}{(T\rho)_{30°C.}}$

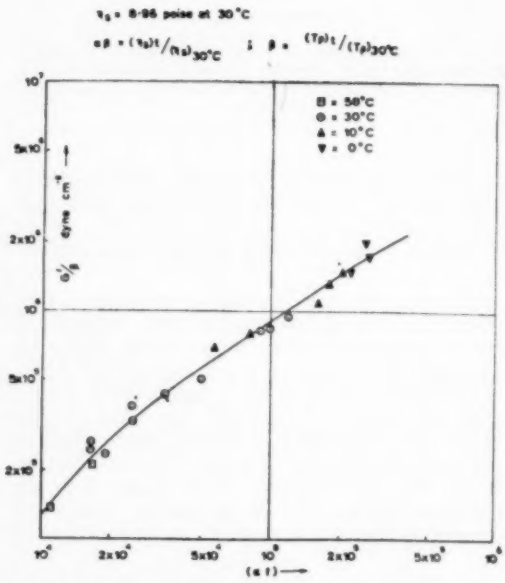

Fig. 12. Plot of shear rigidity modulus G'/β versus (αf) for MS 200/1000 cS. dimethyl silicone normalized to 30°C. η_s = 8.96 poise at 30°C. $\alpha\beta = (\eta_s)_t/(\eta_s)_{30°C.}$; $\beta = \frac{(T\rho)_t}{(T\rho)_{30°C.}}$

interface. The wave is polarized so that the shear displacement is parallel to the plane of the interface. Briefly the principles of the measuring technique are as follows. If a shear wave is reflected at a solid/liquid interface, at normal incidence with the direction of vibration parallel to the interface, the complex reflection coefficient for the shear stress is given by:

$$R \exp[-j(\pi + \theta)] = (Z_L - Z_S)/(Z_S + Z_L), \qquad (14)$$

where Z_S and Z_L are the shear mechanical impedances of the solid and liquid, respectively. In general Z_S is much greater than Z_L and hence the value of R is relatively insensitive to the value of Z_L and is close to unity. An enhanced effect can be obtained by inclining the incident wave at an angle $\varnothing$ to the normal as shown in Fig. 13. Making certain approximations,

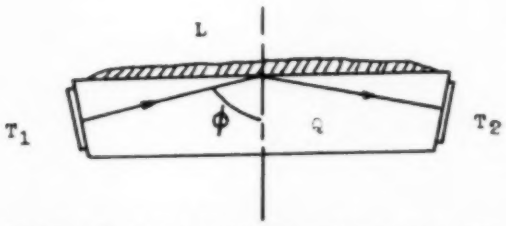

Fig. 13. Reflection of an incident shear wave at a solid-liquid interface. Q = fused quartz bar of rectangular cross-section; L = liquid film; T_1, T_2 = B.T.-cut quartz crystals attached to the inclined ends of the fused quartz bar.

which are valid for all the liquids used, this is equivalent to multiplying the liquid impedance by sec. $\varnothing$ in the expression (14) for the reflection coefficient (O'Neill, 1959) giving:

$$Z_L = R_L + iX_L$$

$$= Z_S \cos \varnothing \left\{ \frac{1 - R^2 + 2jR \sin \varnothing}{1 + R^2 + 2R \cos \varnothing} \right\} \qquad (15)$$

The bar of fused quartz used in the experiments is 7·65 cm. long × 1·6 cm. × 2·15 cm. and $\varnothing$ is approximately 77°. Rectangular B.T. or B.C.-cut quartz crystal plates are 'cold welded' to each end of the fused quartz bar by causing indium films deposited on the crystal and on the end of the bar to coalesce under pressure at a temperature just below the melting point of indium; each film is approximately 10,000 Å thick. The transducers are resonant in the thickness-shear mode of vibration at 3 or 6 Mc/s. and can be driven at any selected odd harmonic. Their positioning is such that on excitation a shear wave is propagated towards the reflecting surface with the shear motion parallel to the surface.

When the shear wave is reflected from a quartz/air interface, R = 1 and θ is zero.

On applying a liquid to the fused quartz, R is reduced and θ increased. In order to determine the resistive and reactive components of Z_L, and hence the components G′ and G″ of the shear modulus, the electrical problem is to measure the changes in amplitude, R, and phase, θ, of the reflected wave arising from the application of a liquid to the fused quartz bar: Z_L is then evaluated from equation (15) using the known value of Z_S (Mason *et al.* 1949).

A schematic diagram of the system is shown in Fig. 14. A transmitter is pulsed at intervals of about 1/200 sec. to provide a modulated pulse of high frequency oscillations of some 5 to 10 μsec. duration. This output pulse is applied to the transmitting transducer and after propagation through the bar the shear wave is received by a second transducer at the opposite end. The duration of the transmitted pulse is less than the time of travel through the bar.

In order to measure the amplitude of the received signal a comparison oscillator of the same frequency is pulsed a short time after the main transmitter; the output is passed through an attenuator and the two signals detected and displayed on the oscilloscope. The time delay is varied so that the received and comparison pulses are displayed side by side. These may then be adjusted to equal height by use of the 'comparison' attenuator. On applying a liquid to the quartz bar, the decrease in amplitude of the received signal is determined by the change in setting of the attenuator needed to re-equalize the two pulses.

For the measurement of phase, a reference signal is derived from an identical quartz bar used as a fixed delay line. The output of this bar is passed through a variable delay line and a variable attenuator, and added to the main signal. By adjustment of the variable line and the attenuator the reference signal is made equal in amplitude and opposite in phase to the main signal, which it therefore cancels. On applying a liquid to the main bar the phase change is determined from the change in setting of the delay-line needed to maintain cancellation. This variable delay-line consists of two parts, a number of fixed lengths of precision coaxial cable in multiples of 10 cm., which may be put into circuit as required, and a short telescopic line for fine adjustment. For convenience the telescopic line is connected in the main channel in series with a 20 db. attenuator to maintain its characteristic impedance and phase linearity; the attenuator may be removed for the amplitude measurement.

Typical measured quantities are 3 db. of amplitude change and 5° phase change (the latter corresponding to about 5 cm. of delay cable at 60 Mc/s.). In the absence of relaxations, liquids of static viscosity greater than about 1 poise can be measured with reasonable accuracy. As the effect of relaxation is, in general, to reduce the measured quantities, it is the effective dynamic viscosity (and elasticity) at a given frequency which is important in deciding whether a measurement can be made with useful accuracy.

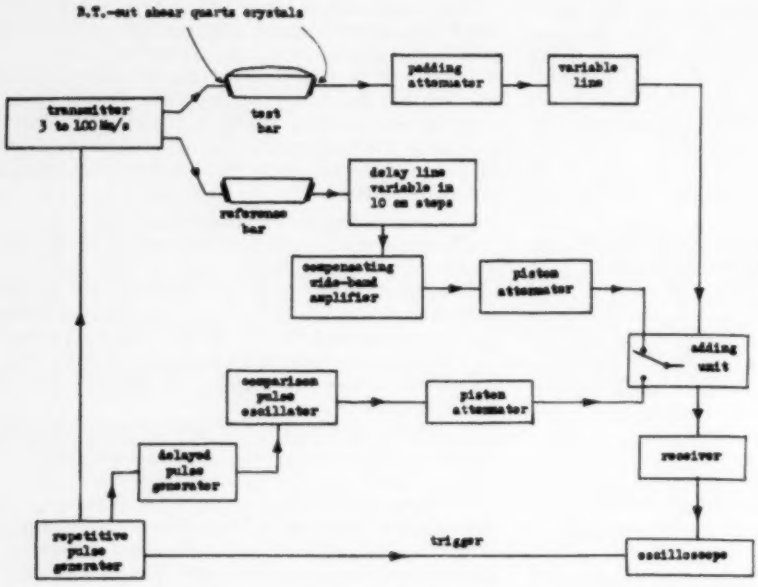

Fig. 14. Schematic diagram of the measuring system for the inclined incidence technique: 3 to 100 Mc/s. (Barlow and Lamb 1959).

The quantities to be measured are increased by taking measurements on signals which have undergone several reflections at the interface: the extent of such improvements in sensitivity is usually limited to a factor of about 3 to 7 (depending on frequency) by the rapid deterioration of the signal to noise ratio with increase in echo number.

A shear wave at normal incidence on a solid/liquid boundary is employed for measurements at high pressures where the system described above is inconvenient. A fused quartz bar of circular cross-section with parallel ends is used and it is then feasible to measure only the amplitude of successive reflections and not the phase change upon reflection. The resistive part of the shear mechanical impedance can then be determined by means of certain approximations which can be shown to be valid in practice. A similar 'normal incidence' technique is also used for measurements at low temperatures where the oil in question is in its 'glassy' state, and for measurements at frequencies above 100 Mc/s. where it becomes increasingly difficult to measure the phase change on reflection.

The accuracy with which R_L and X_L (equation 15) can be determined is governed by the precision with which changes of amplitude and phase can be measured. Both of these measurements are limited by the signal to noise levels and stability of the electrical system.

Amplitude changes can be measured to within $\pm$ 0·05 db., corresponding to $\pm$ 500 Ohm in R_L for the oblique incidence method and $\pm$ 2,400 Ohm with normal incidence. Relative phase shifts are measured only for oblique incidence and can be determined to within $\pm$ 0·33° at 6 Mc/s., equivalent approximately to $\pm$ 500 Ohm in X_L; corresponding figures at 78 Mc/s. are approximately twice these values. Small differences in temperature in the acoustic system can cause appreciable errors in the phase change, expecially at high frequencies, and to a lesser extent in the amplitude change. Typical results obtained by this technique for an oil of medium viscosity index are shown plotted on a 'universal curve' in Fig. 15 (Barlow and Lamb, 1959).

Experimental System Using a Normal Incidence Technique in the Frequency Range 500 to 1,500 Mc/s.

In the frequency range of 500 to 1,500 Mc/s. a normal incidence method is used, and at present measurements are restricted to a determination of the magnitude only of the reflection coefficient. Neglect of the imaginary component of the reflection coefficient introduces very little error in the calculation of the real part of the shear mechanical impedance of the liquid, but gives no indication of the value of the shear reactance.

The main difference between this system and the one described in the previous section is that above about 200 Mc/s. it is very difficult to achieve a good coupling between the transducer and the transmitting medium. In the present system no resonant transducer is used. The transformation from electrical into mechanical energy occurs in an electrical cavity resonator containing a short length of piezoelectric quartz rod. The cavity operates in a quarter wavelength mode and is continuously tunable from 500 to 1,500 Mc/s. by moving a plunger through a slot along the length of the centre post. Two rotateable coupling loops are fixed to the plunger. A quartz rod 15 mm. diameter and 15 mm. long is inserted into the cavity through a hole.

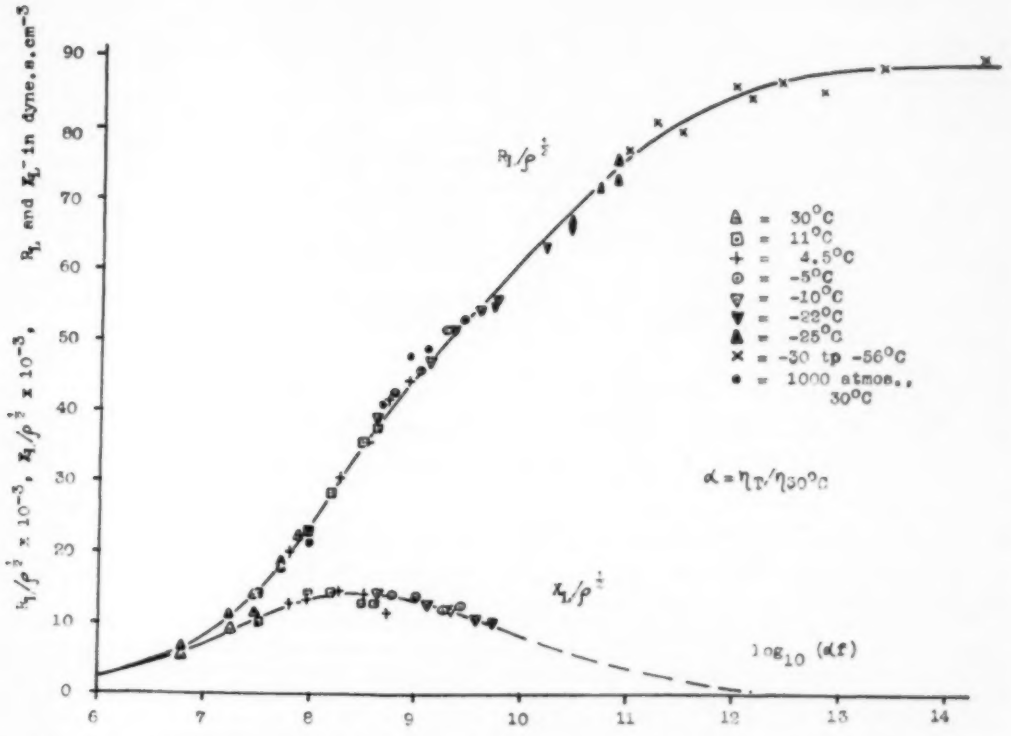

Fig. 15. M.V.I. oil; resistive and reactive components of the shear mechanical impedance. (Barlow and Lamb, 1959).

It has been shown by Bömmel and Dransfeld (1960) that when a piezoelectric material is placed in an alternating electric field mechanical waves are generated at its surface. The fraction of electrical power converted into ultrasonic energy is:

$$F = \left\{ \frac{k^2 Q \lambda A}{2\pi V} \right\} P \qquad (16)$$

where F = mechanical energy
P = electrical power
k = piezoelectric coupling coefficient, a characteristic of the material
Q = quality factor of the cavity
λ = wavelength of the mechanical vibrations
A = area of the generating surface
V = effective volume of the quartz within the cavity

A suitable choice of crystal cut ensures that only one type of vibration is generated and that this vibration is propagated parallel to the rod axis. With the given arrangement of the cavity and quartz a so-called rotated Y-cut is needed to generate pure shear waves, and the condition of parallel propagation requires an angle of rotation of either $-59°$ or $+31°$. The first is a BC and the second an AC-quartz rod. The BC rod is the more suitable since it propagates the shear waves with less attenuation.

The travelling wave generated at the surface of the quartz rod inside the cavity is reflected at the opposite surface; the time for a single transit being about three microseconds. This to-and-fro propagation proceeds until the whole of the energy is absorbed; the cavity is re-energized each time the wave is reflected from the inside surface of the rod. The reconversion from mechanical to electrical energy occurs with the efficiency given by equation (16).

The numerical value of conversion efficiency is of the order of 10^{-3} which implies that the first received pulse would be 10^{-6} times the transmitter pulse even with an attenuation-free medium. A transmitter pulse of high energy is therefore required so that the received pulses are sufficiently above the noise level. The experimental system is shown in Fig. 16. The trigger generator triggers the hydrogen thyratron pulse modulator to produce a pulse of 4 kV. amplitude and 2μs duration. This is fed to the anode of a continuously tunable coaxial line microwave triode oscillator. The output of the oscillator, a train of 500 to 1,500 Mc/s. oscillations

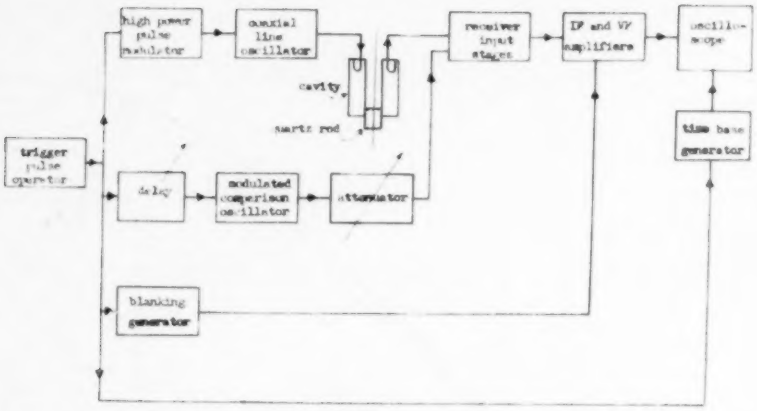

Fig. 16. Schematic diagram of the measuring system for the normal incidence technique: 500 to 1,500 Mc/s.

of 2μs. duration, is coupled to the cavity by coaxial leads and a coupling loop.

The output from the cavity is connected to the input stage of the receiver which has a specially designed thermionic mixer stage capable of withstanding the large transmitting pulse power. The receiver operates on the heterodyne principle and has a wide (10 Mc/s.) bandwidth to prevent distortion of the 2μs. pulses. The I.F. preamplifier is blanked during the transmitter pulse to prevent overloading, so that it is able to receive the first echo. After demodulation the signal is displayed on an oscilloscope.

The trigger generator also triggers a variable delay circuit, which produces a delayed modulating signal for the comparison signal generator. The comparison signal is taken through a variable piston attenuator to the receiver. Measurements are taken by recording the amplitudes of each successive pulse, firstly with the outside end of the rod in air and secondly with the end of the rod immersed in a sample of the liquid under test. This is done by positioning the comparison pulse between two received pulses and adjusting the height of the comparison pulse by means of the piston attenuator until it is the same height as the received pulse.

The liquid is kept as a drop on a circular microscopic slide placed very near to the outside surface of the quartz, and is applied by pneumatic means before the second part of the measurement. This arrangement ensures that the liquid covers the whole of the surface by capillary action, and the electrical characteristics of the cavity are not altered when the liquid is applied.

The difference in attenuator reading between the two sets of measurements is plotted against the echo number giving a straight line. Over 20 pulses are measured at 500 Mc/s. The slope of the line gives the magnitude of the reflection coefficient.

As an improvement it is intended to use two identical cavities separated and fed through a coaxial hybrid ring, so that they may be balanced against each other to give zero output for any particular echo. The effects of applying a liquid to the quartz rod in one cavity may be compensated by the insertion of phase shift and attenuation into the other branch to restore cancellation. With this arrangement it should be possible to determine both components of the shear mechanical impedance of the liquid.

Shear Impedance Measurements of Liquids at 3,000 Mc/s. Considerable progress has been made in developing a measuring system based on the method described in Section 4 and operating at a fixed frequency of 3,000 Mc/s. Owing to the very great attenuation in the quartz crystal it is necessary to reduce the path length to a few millimetres. The transit time in the quartz is about 1 μsec. and this leads to a compromise between conflicting requirements of large transmitted power, short pulse length (and hence a wide bandwidth in the receiving system) and rapid recovery of the receiver with its associated protective device. In practice up to 10 received echos have been observed using a transmitted pulse power of 0·25 MW., a pulse length of 0·15 μsec. and a transit time of 1 μsec.

The quartz crystal must be ground and polished to optical limits of accuracy since the wavelength in the quartz is about 1μ. The electrical system is essentially a waveguide version of the system shown in Fig. 16, and on the basis of present progress it is hoped to make measurements on liquids using this technique in the near future.

ACKNOWLEDGMENTS.—*This work has been supported by a research contract from the National Engineering Laboratory of the Department of Scientific and Industrial Research. We are grateful for this assistance and we appreciate the continued interest of Dr. L. Grunberg of N.E.L. in this work.*

This research has been carried out in the Electrical Engineering Department, Imperial College and is being continued at the University of Glasgow. We also wish to acknowledge the benefit which we have derived from the pioneering work in this field carried out by Dr. W. P. Mason and his colleagues at the Bell Telephone Laboratories.

Our thanks are due to Imperial Chemical Industries Ltd., for items of equipment generously provided under their apparatus-on-loan scheme, for this research.

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The British Laboratory Ware Association

The scientist and technician are well aware of the multiplicity of apparatus and materials in general demand, but many buyers and users may not know how their requirements can best be met. The 50 or so members of the British Laboratory Ware Association throughout the United Kingdom provide the link between many manufacturers of specialist apparatus and materials and the laboratory itself. The member firms include the majority of old-established laboratory furnishers, some of whom supply products of their own manufacture; thus it is that the B.L.W.A. member holding stocks of a great variety of items for immediate delivery and providing staff with specialized knowledge can offer assistance when needed. Also it is by relieving manufacturers of many deliveries and the additional cost of dealing with thousands of accounts that the B.L.W.A. member plays an important role in maintaining an economic and efficient distribution to users. It should, however, be emphasized that the Association exercises no price control over its members' price structures.

The association was formed immediately after the outbreak of war in 1914 with the object of stimulating production in the United Kingdom of laboratory requisites which up till then had been produced mainly in Germany and Bohemia.

Over the years B.L.W.A. members have played an important part in interpreting the requirements of scientists and technicians and in initiating standardization and by co-operating with the British Standards Institution and other bodies, the association being represented on the Industry Standards Committee and on a number of sub-committees of the Institution. The B.L.W.A. membership includes both manufacturers of specialist apparatus, stockists and distributors.

Recent British Standards

pH Scale (B.S. 1647:1961)

The use of pH as a measure of acidity and alkalinity has long been familiar in science and industry. Originally, pH was intended to denote $-\log_{10} C_H$, where C_H is the hydrogen ion concentration, but it is now realized that this quantity is not measurable. In fact, if pH is to be a measurable quantity it cannot have any exact simple fundamental significance. It is therefore necessary to define pH in terms of the method of measurement, and since more than one definition is possible on this basis there is need for a standard pH scale.

In B.S. 1647, (price 4s. 6d.) which was first published in 1950 as a result of an initiative by the Royal Society, the plan adopted is to define the difference in pH between two solutions in terms of a specified electrometric measurement and to complete the definition by assigning a value of pH at each temperature to one chosen solution, termed the *primary standard*. The definition applies to aqueous solutions within certain limits of temperature, and is intended to ensure that, when different workers refer to a stated measured value of pH, they mean the same thing within $\pm$ 0·005. Within this degree of accuracy there is no practical difference between the British Standard pH scale and the scale adopted by the National Bureau of Standards (U.S.A.), although the latter specifies four standard solutions instead of one. The primary standard specified in B.S. 1647 is a 0·05 molar solution of potassium hydrogen phthalate.

No basic change has been made in revising this British Standard, but its usefulness has been increased by extending the range of temperature covered. Originally 0—60°C., this range is now 0—95°C. Corresponding extensions have been made to the relevant tables in the Appendices.

As in the previous edition, recommendations for the use and calibration of the glass electrode are included, together with information on the pH values of aqueous solutions suitable for this purpose. It has not been found possible to make more than one addition to the list of these solutions, the pH values of which must be known reliably to an accuracy of $\pm$ 0·005, and the desirability of further additions is emphasized in the text.

The Sampling and Analysis of Tin and Tin Alloys

The British Standards Institution has recently published the tenth part of a standard designed to cover all methods of sampling and analysing tin and tin alloys.

The latest publication (B.S.3338: Part 10, price 3s.) concerns the photometric method of determining the amount of iron in ingot tin, tin-lead solders and white metal bearing alloys. It specifies the reagents required and recommends methods of sampling and procedure for the determination of an iron content of up to 0·03 per cent.

Methods of Testing Synthetic Rubber Latices

Methods of sampling and testing synthetic rubber latices do not, in all cases, agree with methods used to examine natural rubber latices. Because of this, the British Standards Institution has just issued B.S. 3397 (price 4s.) which deals with synthetic rubber.

At the present moment the standard covers only methods of sampling and determining total solids, coagulum, pH and surface tension. However, work is continuing in the preparation of other standard methods, and these will be issued as addenda to the present document as and when they become available.

Methods of Testing Vulcanized Rubber

Two new methods of test for the determination of rubber-to-metal bond strength are described in the newly issued part A21 of B.S. 903 (price 3s.).

The first method covers rubber which is assembled to two metal plates, and the other deals with rubber assembled to one metal plate. The publication also gives details of the test piece, apparatus, preparation of test piece, procedure, and method of operating.

Copies of these Standards may be obtained from the British Standards Institution, Sales Branch, 2 Park Street, London, W.1.

A RELIABLE METHOD OF DETERMINING EXTINCTION ANGLES OF STREAMING BIREFRINGENT LIQUIDS

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Central Laboratory T.N.O. Delft, the Netherlands

Abstract

When streaming birefringence is investigated with the aid of a Couette type rotor unit, parasitic birefringence of window-glasses may cause systematic deviations of extinction positions at low velocity gradients. This paper describes a compensation method which largely eliminates influence of a weak, homogeneous and time independent proper double refraction of the window-glasses. As a prerequisite, a novel type of window, construction details of which will be published elsewhere, was developed. It allows reliable sealing of the rotor unit without giving rise to the generation of an unhomogeneous additive double refraction of strain in the window-glasses. This is achieved by avoiding cementing or squeezing between packing rings.

Introduction

A liquid containing anisotropic molecules becomes birefringent in a field of flow. This holds especially for solutions of macromolecular substances. As is well-known, in the rotation viscometer a type of laminar flow, which is suitable for the investigation of streaming birefringence, is generated. If the width of the annular gap between rotating and stationary cylinder is small compared with the diameters of the cylinders, the velocity gradient generated in the liquid which fills the annular gap, becomes practically independent of the distance from the wall. As a consequence, a practically uniform streaming birefringence is created.

The light beam required for measuring the streaming birefringence, passes successively through polarizer, annular gap (in axial direction) and analyzer. Polarizer and analyzer are mounted in crossed position, i.e. in a fixed position with respect to each other, but they are allowed to revolve together for the purpose of determining the extinction positions. By introducing a suitable compensator into the light beam, e.g. between annular gap and analyzer, the phase difference generated by the birefringent streaming liquid can be measured.

In general, the extinction angle is defined by the acute angle which is made between the direction of flow (i.e. tangential direction of the cylinders) and either of the two extinction directions. When the extinction angle thus defined, is plotted against the velocity gradient, a monotonously descending curve starting from 45 degrees at the ordinate axis, is obtained. With low molecular liquids, however, the deviation from 45 degrees remains immeasurably small within the experimentally obtainable range of velocity gradients. As is well-known, this range is limited by the onset of turbulence. With solutions of macromolecular substance, on the contrary, the decrease of the extinction angle curve can be considerable. From the initial slope of this curve valuable information on the structure of the solute is obtained (Cerf, 1959).

Measuring points at low velocity gradients become important when the extinction angle curve is bent even at low gradients. Unfortunately this often happens as Leray (1958) pointed out.

As with a low velocity gradient the corresponding deviation from 45 degrees is, in general, also small, a high absolute accuracy of determining the extinction position is required to make the relative error of the initial slope tolerable. The problem is rendered even more difficult by the fact that at a low velocity gradient also the streaming birefringence is small.

Fig. 1 serves for illustration. It shows extinction angle curves obtained with solutions of polystyrene (Montecatini, $M_w = 300{,}000$) in monobromobenzene. The measurements were carried out with an apparatus described previously (Janeschitz-Kriegl, 1960). With the aid of this apparatus extinction positions can be determined by a visual method based on astigmatic optics. Down to a double refraction Δn of about 2×10^{-8} the error for a single reading is, independent of the magnitude of birefringence, equal to about 0·1 degree. For all but one curve of Fig. 1 systematic deviations may be noticed from the expected course which is extrapolated to an ordinate value of 45 degrees with decreasing velocity gradient. The figures near the curves give the concentrations of the solutions in grams per hundred millilitres. The lower part of Fig. 1 gives the corresponding birefringence curves, which enable one to form an idea about the magnitude of the streaming birefringence effects at which the deviations occur in our apparatus. Birefringence was measured with the aid of monochromatic light of wave length $\lambda = 548$ mμ. Deviations of the same order of magnitude are shown by extinction angle curves previously published by the author (Janeschitz-Kriegl, 1959, 1960).

Evidence was also given that in our apparatus the described deviations were caused by the (very weak) birefringence of the glass windows of the rotor unit. By turning the windows deviations could be influenced arbitrarily within certain limits, and this experience formed the starting point of the present investigation. For the windows cover glasses were used. These glasses were cemented with the aid of a mixture of water-glass and glycerol on revolving glass fittings. For every run new cover glasses were cemented.

The double refraction of strain of a window is built up from two independent contributions, viz. the proper

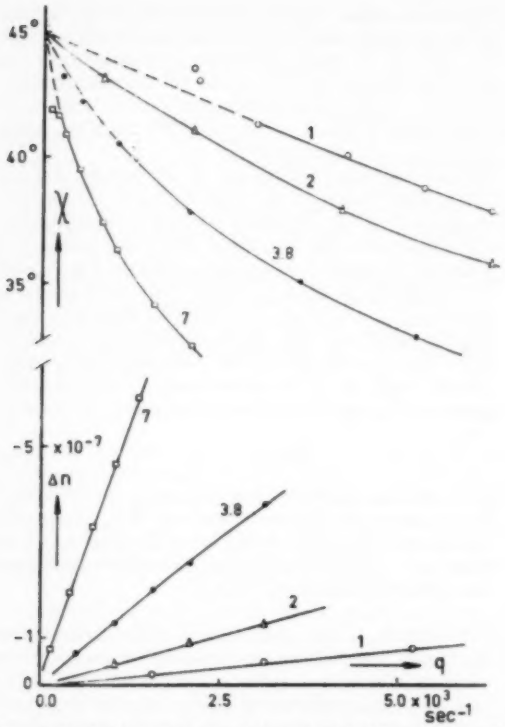


Fig. 1. Extinction angle χ and streaming birefringence Δn of solutions of polystyrene (M_w = 300,000; M_n = 190,000) in bromo-benzene. Figures near the curves give concentrations in g/100 cm³. q gives the velocity gradients applied. In the performance of these measurements cemented windows were used.

double refraction of strain of the unloaded glass and the additional double refraction of strain which is produced by the sealing manipulation (cementing or squeezing between packing rings). It is our experience that every type of glass useful for the manufacture of windows possesses a slight proper double refraction. This double refraction, however, has two important advantageous features. Firstly, it is practically independent of time. Secondly, it is, at least with optical glasses, sufficiently homogeneous for making windows with practically uniform double refraction. The additional double refraction of strain which is generated by the sealing procedure, possesses neither of the two mentioned advantages.

Application of these experiences was facilitated by the invention of a new window design by Mrs. E. van Kuik-van Meerten in our laboratory. This type of window allows a secure sealing of the rotor unit without making use of cementing or squeezing. In this way, the above mentioned additional double refraction of strain can be carefully avoided. The description of this window will be published elsewhere.

A description will now be given of a method which admits compensation for the disturbing influence of weak, but time-independent window birefringence. This compensation can be adjusted once for all, and holds as long as the same pair of windows is used.

Theory

In the range of velocity gradients in which the influence of double refraction of the windows is noticeable, all participant double refractions are extremely small. Consequently, for the ellipticity of the wave which reaches the analyzer, the following simple equation holds:

$$2\psi^* = \Delta \sin 2(\beta - \chi) + \delta_w \sin 2(\beta - \alpha_w) + \delta_p \sin 2(\beta - \alpha_p) + \delta_c \sin 2(\beta - \alpha_c) \ldots (1)$$

ψ^* . . . ellipticity of the wave at the exit from the rotor unit.

β . . . azimuth of the polarizer.

The phase differences δ and the azimuths α belong to the following double refractions:

Δ, χ . . . streaming birefringence

δ_w, α_w . . . double refraction of the windows taken together (explanation below)

δ_p, α_p . . . double refraction of strain of the polarizing prisms (together)

δ_c, α_c . . . double refraction of an introduced compensator plate.

The azimuth of the wave is not altered to any extent during passing the double refractive media and remains practically equal to that of the polarizer. By making the ellipticity ψ^* equal to zero, the azimuths of the polarizer at which extinction occurs, are found. Due to the additivity of contributions, the two windows can be considered to form one double refractive medium as long as they are not turned with respect to each other. The same holds for the double refractions (of strain) of the polarizing prisms.

For the azimuth χ of the streaming birefringence one may take the extinction angle as defined above. For the remaining azimuths also the direction of flow may be chosen as direction of reference, The compensator plate consists of a weakly birefringent glass plate which is mounted revolving on the fitting of the polarizer. The double refraction of this glass plate must be sufficiently homogeneous and at least somewhat larger than the sum of the double refractions of windows and polarizing prisms. Polarizer and analyzer are used in crossed position. From these arrangements it follows that:

$$\beta - \alpha_p = \gamma = \text{constant}$$
$$\beta - \alpha_c = \varepsilon = \text{adjustable} \quad \ldots\ldots (2)$$

Furthermore it is assumed that the (correct) extinction angle χ of the streaming birefringence under consideration does not deviate very much from 45 degrees (cf. introduction). Because only small deviations from this correct extinction direction are expected, for the azimuth β at which ψ^* vanishes (i.e. the experimentally determined extinction angle) a similar assumption is made, i.e.:

$$\chi = \pi/4 - \psi \qquad \psi \ll 1$$
$$\beta = \pi/4 - \xi \qquad \xi \ll 1 \quad \ldots\ldots\ldots (3)$$

The value of β at which ψ* vanishes follows from equation (1). With the simplifications of equations (2) and (3) the following expression results:

$$\xi = \pi/4 - \beta = \frac{\psi}{1 + (\delta_w/\Delta) \sin 2\alpha_w} + \frac{\delta_w \cos 2\alpha_w + \delta_p \sin 2\gamma + \delta_c \sin 2\varepsilon}{2(\Delta + \delta_w \sin 2\alpha_w)} \quad \ldots (4)$$

Equation (4) contains a sufficient number of independent variables to permit the following conditions to occur simultaneously:

$$\sin 2\alpha_w = 0 \text{ (by revolving the windows)} \quad \ldots (5)$$

$$\delta_w \cos 2\alpha_w + \delta_p \sin 2\gamma + \delta_c \sin 2\varepsilon = 0$$
(by adjusting the compensator plate) . . . (6)

In this way, independent of the magnitude of the double refraction of strain of the windows, the desired result is obtained, i.e.:

$$\xi = \psi \quad \ldots\ldots\ldots (7)$$

The azimuths α_w and ε, however, are not explicitly known. For this reason the following procedure of adjustment is recommended:

Before putting the rotor unit into the light beam, the strains of the crossed polarizing prisms are compensated for as much as possible by turning the compensator plate until optimum darkness is obtained*. Then the following relation holds:

$$\delta_p \sin 2\gamma + \delta_c \sin 2\varepsilon \ll \delta_w \quad \ldots\ldots (8)$$

After reaching this situation, the rotor unit containing the complete window arrangement and a low molecular weight test liquid (e.g. α-methyl naphthalene or α-bromo naphthalene) is put in the light beam. If now a high velocity gradient is applied to the test liquid, the double refraction of the windows becomes negligible compared with the streaming birefringence created. Consequently, by determining the extinction directions at a high velocity gradient, the exact position of the direction at an angle of 45 degrees with the direction of flow is found. (As mentioned in the introduction, the extinction angle of a low molecular weight liquid does not deviate from 45 degrees).

If the streaming birefringence of the test liquid is reduced to such an extent that the double refraction of the windows is no longer negligible, a deviation ξ of the observed extinction position from the direction at 45 degrees is found. By revolving the more birefringent window, however, this deviation can always be made to vanish. If the velocity gradient is further reduced, this adjustment of the window (aiming at $\xi = 0$) becomes more and more sensitive. For the test liquid one has $\chi = \pi/4$ or $\psi = 0$. Consequently it follows from the equations (4) and (8) that by the adjustment described $\cos 2\alpha_w$ is made practically equal to zero. Thus α_w is made nearly equal to 45 degrees. This may be called the first step of the adjustment.

From equation (5), however, it can be seen that the preferential values of α_w are zero and 90 degrees, respectively. As the 45-degree position is established by the adjustment described above, one of the preferential positions is reached by revolving both windows over 45 degrees in the same direction. Both windows must be turned as the angle α_w refers to their combined double refraction. The angle of revolution must be read from graduations at the windows. By this procedure, which forms the second step of the adjustment proposed, equation (5) is satisfied as well as possible, i.e.:

$$\sin 2\alpha_w \ll 1 \quad \ldots\ldots\ldots (5a)$$

At the same time, however, the deviation ξ increases to the maximum value: Indeed, $\cos 2\alpha_w$ attains the value one. As the third step, the deviation ξ can be made zero again by readjusting the compensator plate (i.e. until equation (6) is satisfied).

Finally, as a completion to the more mathematical derivation given above, the physical background of the method of adjustment will be elucidated. If the extinction directions of the two windows which are considered as one birefringent medium, are brought into positions parallel and perpendicular to the direction of flow, at an azimuth $\beta = 45°$ of the polarizer, a maximum ellipticity is created by the windows. However, by the compensator plate mounted to the polarizer the said ellipticity is compensated. This compensation remains valid also in the neighbourhood of the maximum: Indeed, at an azimuth $\beta = 41°$ the ellipticity created by the windows is decreased only by 1 per cent.

As an additional remark it may be mentioned that, in principle, a set of three birefringent plates with low double refraction of nearly the same magnitude can be so adjusted as to make the ellipticity vanish identically for all values of the azimuth β of the polarizer. It can be seen from equation (1) that the following relationships must be satisfied:

$$\delta_1^2 + \delta_2^2 + 2\delta_1\delta_2 \cos 2(\alpha_1 - \alpha_2) = \delta_3^2 \quad (9)$$

$$\frac{\delta_1 \sin 2\alpha_1 + \delta_2 \sin 2\alpha_2}{\delta_1 \cos 2\alpha_1 + \delta_2 \cos 2\alpha_2} = \operatorname{tg} 2\alpha_3 \quad \ldots (10)$$

In this relative position the set of three plates is equivalent to a non-birefringent plate. For our purpose two of these plates may be identified with the windows of the rotor unit, the third plate with a compensator plate fitted, in contrast to the compensator plate used above, to the rotor unit. After a suitable adjustment and fixation of the favourable positions any influence of

*This adjustment is quite sensitive as a sufficient amount of light passes the polarizing prisms as long as the rotor unit is removed.

window birefringence would be eliminated completely. However, with the low double refractions in question a practicable method of adjustment could not be devised.

Experiments

Considering now the experimental part, we were interested especially in the performance of the third step of the adjustment. No details will be given on the performances of the first and second steps (discussed above).

Consequently, it is assumed here that equation (5) (or 5a) was already satisfied with sufficient accuracy. If, moreover, the double refraction of strain of the polarizing prisms is disregarded for convenience, equation (4) reads:

$$\xi - \psi = (1/2\Delta)\,(\delta_w + \delta_c \sin 2\,\varepsilon) \ldots . \quad (4a)$$

From this equation it becomes evident once more that, on condition that equation (5) is satisfied, the deviation $(\xi - \psi)$ no longer depends on whether ψ is equal to zero or not. This deviation is a sinusoidal function of the position ε of the compensator plate if the streaming birefringence is kept constant (Δ = const.).

The position ε of the compensator plate can be read with the aid of a graduation engraved on the rim of its fitting and a nonius attached to the housing of the polarizing prism. Actually, in this way ε is determined except for an arbitrary constant. (The sign of ε is irrelevant in this connection). One reads:

$$\varepsilon^* = \varepsilon + C_1$$

Unfortunately the direction of the polarization of the polarizer can be read only from an arbitrary graduation. Without further knowledge about the method of graduation, no more can be predicted than:

$$\pm\,\beta^* = \beta + C_2$$

However, the value of β^*_{45} which corresponds with the direction at 45 degrees with stream lines ($\beta = \pi/4$), can be found very easily. The well-known method of applying both directions of revolution to the rotor is used. If the number of revolutions is kept the same, then with the change of the direction of revolution in equation (4a) only the sign of Δ, and with it the sign of $\xi - \psi$, is changed.* As a consequence, for the two directions of revolution, two symmetrical curves yielding β^* as sinusoidal functions of ε^*, are expected. One of these curves must coincide with the other after reflexion at the horizontal line passing through the points of intersection of the curves. The common ordinate of the points of intersection furnishes the required value of β^*_{45}.

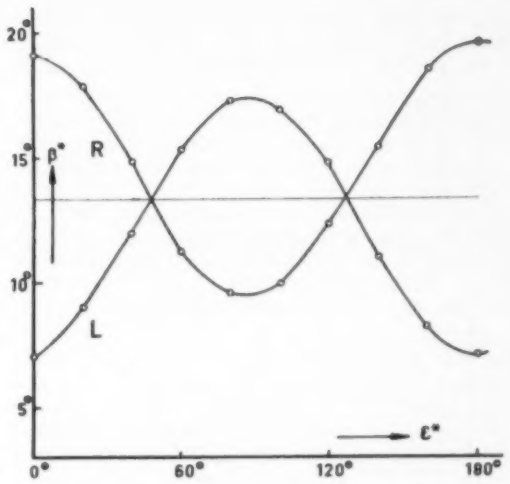


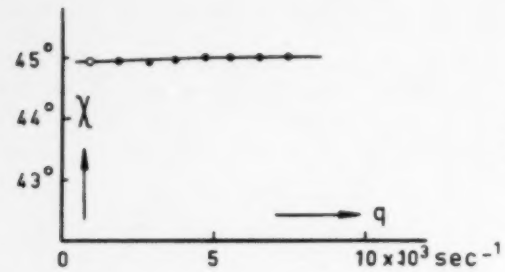
Fig. 2. Illustration of the method of finding the compensation positions ε^*_1 and ε^*_2 of the compensator plate. β^* gives the readings at the graduation of the polarizer at which extinction occurs with various positions ε^* of the compensator plate. Both directions of revolution (L and R) are applied at a velocity gradient of q = 1,860 sec^{-1}. α-bromo-naphthalene was used as a test liquid.

As *Fig. 2* shows, the expected behaviour is actually found. As a low-molecular weight test liquid ($\psi = 0$), α-bromo-naphthalene was used. The measurements were carried out at a velocity gradient q = 1,860 sec^{-1}. The ordinate of the points of intersection is found to be $\beta^*_{45} = 13{\cdot}4°$. The two (equivalent) compensation positions of the compensator plate are equal to the abscissa values of the points of intersection ($\varepsilon^*_1 = 48°$, $\varepsilon^*_2 = 127°$). The quotation of the directions of revolution L and R may serve here only for discrimination. It will be sufficient for the following to state that with direction L in our apparatus the β^* values increase together with the positive ξ-values.*

Using the compensation position $\varepsilon^*_1 = 48°$, the extinction angle of α-bromo-naphthalene is determined practically without disturbance down to the (relatively small) velocity gradient q = 1,860 sec^{-1} at which the adjustment was carried out. This is shown in *Fig. 3*. In this figure the open circle indicates a measuring point found even at a smaller velocity gradient. Fig. 3 also contains the streaming birefringence of α-bromo-naphthalene as a function of the velocity gradient. The birefringence is calculated from the phase difference Δ with the well-known equation:

*The definitions $\chi \leqslant \pi/4$ and $-\psi \geqslant 0$, respectively, (cf. introduction) are dropped here for practical reasons. Actually, observing this definition, Δ has a definite sign depending merely on the properties of the streaming liquid under consideration. The positive sign holds, if the refractive index which is obtained for a direction of polarization parallel to the main tension, is the larger one. This direction of main tension in streaming liquid, coincides with that extinction direction which makes the acute angle L = ('extinction angle') with the direction of flow. In contrast to this definition given on physical grounds, the practical method proposed here leads to a change of the sign of Δ with the change of the direction of flow and, at the same time, to a transition from an acute extinction angle ($\psi \geqslant 0$) to an obtuse extinction angle ($\psi \leqslant 0$).

*In other words, for the direction L the original definition of the extinction angle is maintained.



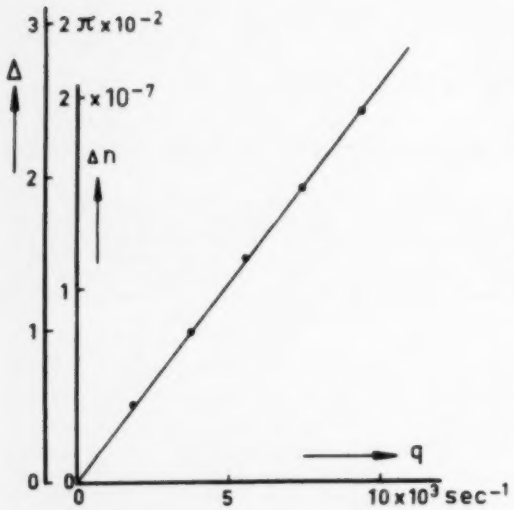


Fig. 3. Extinction angle χ and streaming birefringence Δn of α-bromo-naphthalene as functions of the velocity gradient q. Δ gives the phase difference corresponding with the birefringence Δn. In performing these measurements, the novel windows were used.

$$\Delta n = (\Delta/2\pi)\,(\lambda/D) \quad \ldots\ldots\ldots (11)$$

Δn streaming double refraction
Δ phase difference observed
λ wave length of monochromatic light (λ = 548 mμ) used for the quarter wave compensator*
D height of the cylinders (D = 7 cm.).

From *Fig. 3* a phase difference $\Delta = 0{\cdot}50 \times 2\pi \times 10^{-2}$ can be read for the velocity gradient q = 1,860 sec^{-1}. From Fig. 2 for the maximum of the L-curve a value $\xi_1 = 4{\cdot}0°$ and for the minimum $\xi_2 = -6{\cdot}3°$ is found. With the aid of these values, the scheme (following from equation 4a).

$$\xi_1 = (1/2\Delta)\,(\delta_w + \delta_c)$$
$$\xi_2 = (1/2\Delta)\,(\delta_w - \delta_c)$$

furnishes values for δ_c and δ_w which are tabulated together with Δ in Table 1. (For this calculation the ξ-values, expressed in degrees, first were transformed into arcs).

TABLE 1

$$\Delta = 0{\cdot}50 \times 2\pi \times 10^{-2}$$
$$\delta_c = 0{\cdot}88 \times 2\pi \times 10^{-3}$$
$$\delta_w = -2{\cdot}0 \times 2\pi \times 10^{-4}$$

It is noticed that ξ_1 and ξ_2 are somewhat too large for the conditions expressed in equation (3). However only the order of magnitude is of interest here. From Table I it is noticed that in the direction of flow ($\alpha_w = 0$), the window combination has a smaller index of refraction than in the perpendicular direction (negative sign of δ_w).

From Fig. 2 also the position of $\varepsilon = 0$ can be found. The extremes of the curves lie symmetrically with respect to ε^*_1 and ε^*_2. From this it follows that $\varepsilon = 0$ pertains to a ε^*-value of 42·5°. (This ε^*-value is identical with C_1 of the above equation connecting ε^* and ε).

Furthermore it is interesting to estimate the deviation of the experimental extinction angle in the absence of the compensator plate. For α-bromo-naphthalene and a velocity gradient of q = 1,860 sec^{-1} one obtains with the aid of equation (4a) ($\delta_c = 0$)

$$\xi_L = (\delta_w/2\Delta) = -1{\cdot}1°$$

This means that in Fig. 3 the measuring point at q = 1,860 sec^{-1} would be 1·1° too high. The open circle would even be shifted 2·5° in an upward direction.

Another question arises about the change of shape of the curves of Fig. 2, if equation (5) would no longer be sufficiently satisfied. If only the first step of the proposed adjustment was carried out, for a low molecular weight liquid, equation (4) would read (with $\sin 2\alpha_w = 1$ and $\cos 2\alpha_w = 0$):

$$\xi = [1/2\,(\Delta + \delta_w)]\,\delta_c \sin 2\varepsilon \quad \ldots\ldots (4b)$$

From this equation it follows that the amplitudes will be different for the two directions of revolution ($\pm\Delta$). In our example, however, Δ is still relatively large compared with δ_w. As a consequence the difference of the amplitudes is not very marked, as is shown in *Fig. 4*. In this figure the curves calculated from equation (4b) (with the aid of Table I) are drawn as continuous lines, whereas for comparison the experimental curves of Fig. 2, which both possess the same amplitude, are given as dotted lines.

The conclusion may be drawn that equation (5a) does not give a critical condition, as long as Δ is considerably larger than δ_w.

*Extinction angles are determined with the aid of the practically white light of a high pressure mercury arc. However, for the low phase differences in question, there is no difference between results obtained with white light and with monochromatic light of wavelength λ = 548mμ.

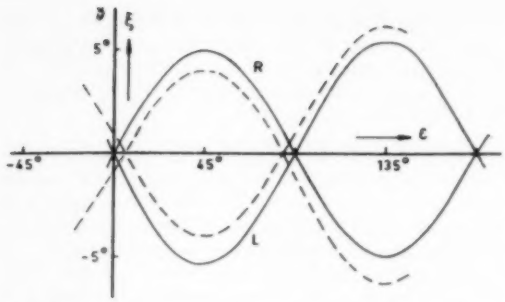


Fig. 4. Representation of the curves expressed by the equations (4a) (dotted lines) and (4b) (full curves). For the calculation the values of Table 1 are used. L corresponds with $+\Delta$, whereas R corresponds with $-\Delta$.

Results and Conclusions

Fig. 5 gives extinction angle curves and streaming birefringence curves as functions of the velocity gradient for dilute solutions of two types of polystyrene in monobromo-benzene. For the determination of these curves the new type of window was used. The figures near the curves give the concentrations in grams per hundred millilitres. The concentrations 0·50, 0·75 and 1·00 concern solutions of a linear fraction of weight average molecular weight $M_w = 410{,}000$ and number average molecular weight $M_n = 270{,}000$. This fraction was kindly provided by S. Olivé and G. Olivé-Henrici (Monsanto Research S.A., Zürich). The concentration 1·5 is that of a solution of 'Polystyrol III, BASF.'. The interpretation of the curves from the point of view of molecular structure will be given elsewhere. Only the reliability of the measuring results will be considered here. It appears to be much better than for the results plotted in Fig. 1. So far it had not been possible to determine, with our apparatus, extinction angle curves of solutions of polystyrenes with comparable molecular weight at such low concentrations as 0·5 and 0·75 gr./100 ml., using monobromo-benzene as a solvent with relatively low viscosity.

The observable inversion of the order of succession of the extinction angle curves with respect to decreasing concentration is merely due to the contribution of the streaming birefringence of the solvent, because this contribution is no longer negligible at low concentrations (cf. lower part of Fig. 5, showing the streaming birefringence of the pure solvent). This statement will be substantiated in a paper to be published elsewhere.

Systematic deviations are observed for the extinction angles determined at velocity gradients $q \approx 900\ \text{sec}^{-1}$. According to the definition of equation (3) (which remains valid for the L-direction of revolution), these deviations are negative ($\xi - \psi < 0$). Since also Δ is negative for the polystyrene solutions, it may be argued from equation (4a) that the value of $\varepsilon^*_1 = 48°$ is somewhat too large. From a quantitative treatment, using Table I and the value $C_1 = 42{\cdot}5°$, it follows that ε^*_1 must be chosen about one degree smaller, in order to obtain a close fit of the experimental points on the drawn extinction angle curve.

However, with the visual determination of extinction angles, application of the described method is limited. Such determinations must be repeated about ten times at a low streaming double refraction of $\Delta n \approx 1 \times 10^{-8}$ to obtain reliable average values. The efficiency of the described method might possibly be enhanced by the use of one of the more sensitive photoelectric methods of determining extinction positions as proposed by B. H. Zimm (1958), by H. Wayland (1959), by H. Wayland and J. Badoz (1960) and by J. Leray and G. Scheibling (1960).

The advantages of the new window design are not restricted to the field of optics. Cementing of the windows, a very troublesome work, is no longer necessary, and possible interactions between cement and solvent become irrelevant. Moreover, work at

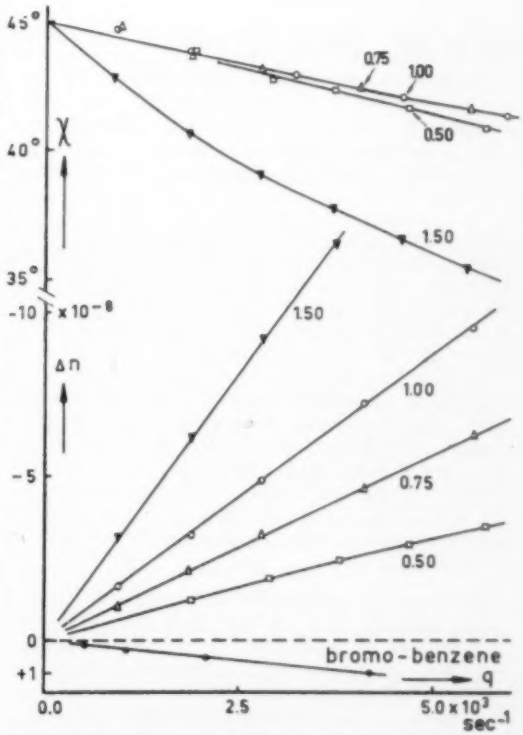


Fig. 5. Extinction angle χ and streaming birefringence Δn as functions of the velocity gradient q for solutions of two polystyrenes in bromo-benzene. The concentrations (given by the figures near the curves) 0.50, 0.75 and 1.00 g/100 cm³ refer to a linear polystrene fraction ($M_w = 410{,}000$; $M_n = 270{,}000$). The concentration of 1.5 g/100 cm³ refers to a technical grade. In carrying out these measurements, the novel windows were used.

elevated temperatures is possible. With suitable handling the service life of the new window is practically unlimited. However, these features will be explained more conveniently in the description of the construction of the windows which will be published elsewhere.

ACKNOWLEDGMENTS.—*The author is indebted to Mrs. E. van Kuik-van Meerten and Miss C. Spijkers for carrying out the measurements.*

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BOOK REVIEWS

Provision of Laboratory Animals for Research, by W. Lane-Petter. Amsterdam: Elsevier, U.K. Distributors: D. Van Nostrand Co., Ltd. 1961. pp. 147 + xii. 20s.

This monograph, as the author emphasizes in his preface, is concerned with the principles involved in the provision of laboratory animals for research. It is addressed primarily to departmental heads and scientific administrators who are concerned with the general problems of how many animals are needed, of what kind, how they can be produced, and how much they will cost. In this sense it is, as sub-titled, a practical guide. The book makes no attempt to deal in detail with the technical problems involved in the day-to-day operation of an established animal house. A number of standard works and original articles are cited in the text and are listed under the headings bibliography and references respectively. An adequate subject index is provided.

The first four chapters are concerned with filling in the background. They deal with utilization of animals, their sources, the likely trends in requirements, and the economics of the provision of laboratory animals. The following five chapters deal with the control of quality in laboratory animals. Separate chapters are devoted to genetics, health, nutrition, physical environment, and the organization of care of the animals. Of the remaining three chapters, one is concerned with the quantitative aspects, with production techniques; one with the function of laboratory animal centres and the final chapter with legal and humane aspects and with public relations.

In general the book is well produced save for the occasional printer's error which has gone undetected. The unfortunate substitution of homozygous for heterozygous with reference to the F_1 genotype in Fig. 4 is a more serious error.

The book without a doubt achieves the objectives set out by the author in the preface, provided that the persons to whom it is addressed bother to read it. In any event the existence of this monograph will hearten those who have struggled with the day-to-day organization of animal houses. Here is a book to be handed to those scientists who habitually expect to obtain so many animals of such and such species in this or that physiological state at a moment's notice.—R. E. MUNFORD.

X-Ray Microscopy, by V. E. Coslett, M.A., Ph.D., and W. C. Nixon, M.A., Ph.D. Cambridge: University Press. 1960. pp. 406 + xiv. 80s.

Much exciting work has been done during the last few years in the no-man's-land which lies between Radiography, Electron Microscopy and X-ray Diffraction. This field is now generally referred to as X-ray Microscopy. In the authors' words, 'X-ray microscopy provides information supplementary to that obtained with the established optical and electron methods. The physical respects in which it differs from them define its special sphere of usefulness: short wavelength, small but specific absorption, and a simple emission spectrum. In addition it shares with electron microscopy, in contrast to the light microscope, the possibility of micro diffraction on crystalline specimens and of stereophotography at high resolution on those of open work structure'. An authoritative review in book form has been lacking until now and the present volume is to be welcomed all the more since it brings together methods and techniques of interest to the metallurgist, the mineralogist, the crystallographer and the biologist.

Successive sections deal with contact microradiography, microscopy by point projection and reflexion X-ray microscopy. Micro analysis by X-ray absorption and emission are then discussed. In each case the principles of the method, the techniques used and some typical results are described. Enough formulae are given to enable the reader to appreciate the advantages and limitations of the different methods, without overburdening the text with mathematical equations. Many of the new techniques originated, or were further developed in the authors' laboratories, but they have successfully resisted the temptation of going into too detailed descriptions of particular arrangements.

The book is recommended alike to those who wish to assess the usefulness of the new techniques for their own problems and to those who want to further their studies of them.—U. W. ARNDT.

STREAMING DOUBLE REFRACTION IN LIQUIDS

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Abstract

The study of the double refraction produced in flowing liquids provides useful information for the determination of molecular size, shape and weight. The quantities required are the birefringence produced and the flow gradient. In this paper the relationship between the birefringence and the molecular polarizability and dimensions is discussed. Details are presented of an apparatus used for this type of work. This consists essentially of two concentric cylinders arranged as in a Couette viscometer with the outer cylinder rotatable to speeds as high as 2,500 r.p.m. Gradients of the order of $4{,}000\pi$ sec^{-1} can be produced. A cooling system is provided to maintain the liquid at a constant temperature. The outer cylinder is provided with a glass bottom so that linearly polarized light can be made to traverse the liquid in the annular gap between the cylinders: on emergence it is analyzed by a suitable optical system. The accuracy of the instrument is discussed and some results on solutions of polystyrene in toluene are presented.

Introduction

When a velocity gradient is established in a liquid or solution, the shearing forces to which the molecules or solute particles are subjected can produce a measurable amount of double refraction. This was first observed by Maxwell (1873). A number of theories have been put forward to explain this effect (Jerrard, 1959). For liquids and dilute solutions it is usually assumed that under the influence of the shearing forces the molecules will orientate and perhaps deform. The orientation will be opposed by the disordering effect of thermal agitation so that finally the molecules will reach a kinematic state of equilibrium in which they spend most of their time lying in a definite direction, in fact along the streamlines. When a light wave is incident on this system of ordered particles the optical behaviour is determined by the induced polarization brought about by the electric field of the incident light wave. For concentrated solutions, in particular polymers, a different mechanism has been suggested (Lodge, 1956). In this it is assumed that a loose network structure extends throughout the solution and that this is deformed by the flow gradient to give rise to stresses. The latter render the liquid birefringent.

Flow gradients can be produced by allowing the liquid to flow through a tube but such a gradient is not constant across the tube and high values cannot be realized. A better method is to enclose the liquid in the annular gap between two concentric cylinders one of which can rotate. With such an arrangement constant and high gradients can be obtained and these conditions

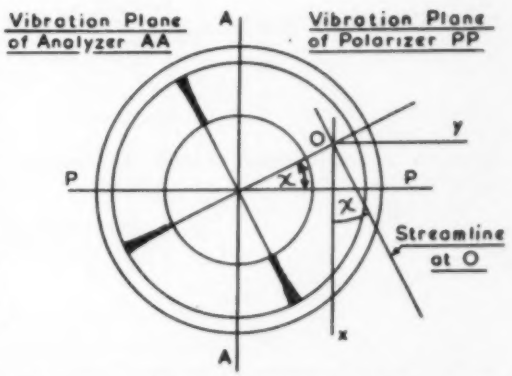

Fig. 1. View of the doubly refracting medium in the annular gap between concentric cylinders.

of stress render the liquid optically biaxial (Jerrard, 1957). If the annular gap is viewed between crossed polarizing devices, so that the light travels parallel to the axes of the cylinders and is thus perpendicular both to the streamlines and to the velocity gradient, the field will appear dark when both cylinders are at rest. When one cylinder rotates the field of view will appear bright everywhere except for four regions equispaced around the gap; they form the arms of a cross called the cross of isocline (Fig. 1). The position of the cross is specified by the 'extinction angle' χ, which is defined as the smaller of the two angles between the vibration planes of the crossed polarizing devices and the arm of the cross. The linearly polarized light beam on passing through a length d of the liquid will be resolved into two linearly polarized components, one vibrating along Ox and the other along Oy. These emerge with a phase difference Δ (in radians) given by $\Delta = 2\pi d\,\Delta n/\lambda$, where Δn is the flow birefringence of the liquid, λ is the wavelength in vacuo. d is measured in the same units as λ. The birefringence Δn is given by $n_x - n_y$ where n_x and n_y are the refractive indices of the medium along Ox and Oy.

Streaming double refraction studies give important information on molecular parameters because χ and Δn are related to the sizes, shapes and polarizabilities of the molecules. It is usual in the case of liquids and colloidal solutions to take as a model for the molecule an ellipsoid of revolution of axial ratio r. Then according to the orientation theories mentioned previously, for liquids (Raman and Krishnan, 1928; Peterlin and Stuart, 1939b)

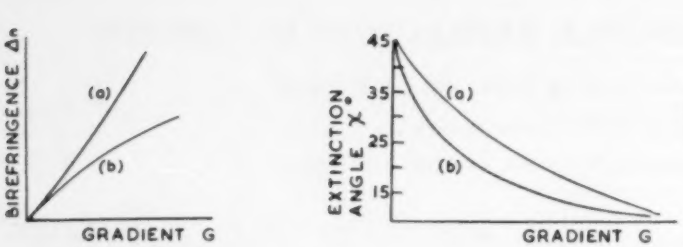

Fig. 2. Variation of birefringence △n and χ with gradient G. (*a*) Soft molecule, (*b*) rigid molecule.

$$\Delta n = f_1[n, (\alpha_1 - \alpha_2), G, b, D]$$
$$\chi = \pi/4$$

whilst for dilute solutions (Peterlin and Stuart, 1939b)

$$\Delta n = f_2[n_s, n_o, (\alpha_1 - \alpha_2), G, b, D]$$

$$\chi = \frac{\pi}{4} - \frac{G}{12D}\left(1 - \frac{G^2}{108D^2}\left(1 + \frac{24b^2}{35}\right)\right)$$

f_1 and f_2 denote two different functions, n, n_s and n_o are the refractive indices of the liquid, solute and solvent respectively, $(\alpha_1 - \alpha_2)$ is the difference between the polarizabilities along the ellipsoid axes, G is the flow gradient. $b = (r^2-1)/(r^2+1)$. D is the rotary diffusion constant which is a measure of the ability of the molecule to rotate and is a function of r and the viscosity η_i of the solution.

For chain molecules the molecular size is often characterized by the mean square of the distance between the ends, i.e. by $\overline{h^2}$ and for dilute solutions

$$\Delta n = f_3[n_o, (\alpha_1 - \alpha_2), G, \overline{h^2}, D]$$

and $$\cot 2\chi = \beta \frac{G}{D} \overline{h^2}.$$

f_3 denotes another function and β is 3 or unity according to whether the molecule is rigid or soft.

The variation of Δn and χ with G predicted from these equations is shown in Fig. 2. When concentrated solutions are considered, Lodge finds that Δn is related to χ, the relationship being

$$\frac{\Delta n}{\bar{n}} = \frac{4\pi}{5}\left(\frac{\overline{n^2}+2}{3\bar{n}}\right)^2 \frac{\alpha_1 - \alpha_2}{kT} \eta_i G \operatorname{cosec} \chi$$

$\bar{n}$ is the mean refractive index of the solution, η_i its viscosity coefficient. k and T are Boltzmann's constant and the absolute temperature respectively.

It is clear then that the quantities requiring measurement are χ, Δn and G for these, with refractive index values, enable b, $\overline{h^2}$, D and the anisotropy of the polarizability $(\alpha_1 - \alpha_2)$ to be determined. It is also desirable to determine η_i under the prevailing conditions of flow.

Apparatus

The apparatus for double refraction studies consists of two concentric cylinders mounted vertically as in a Couette viscometer. The outer cylinder (rotor) is driven with a belt drive connected to a variable speed motor, while the inner one (stator) is suspended with its motion controlled by a torsion wire. It is preferable to rotate the outer cylinder and not the inner cylinder because then turbulence is less likely to occur (Jerrard, 1950). The rotor is surrounded by a cooling jacket for temperature control and its speed of rotation measured by an electronic tachometer (Jerrard and Punnett, 1950, 1951). The cylinder assembly is placed in an optical system so that a linearly polarized light beam can pass through the annular gap between the cylinders into a unit (Jerrard 1954) which enables the double refraction Δ to be measured. Fig. 3 shows the apparatus.

The rotor is made from a solid billet of stainless steel and the whole of its inside surface ground parallel to 0·0001 in. It is mounted on two ball journal races in a brass housing. The bottom is closed with a strain free glass disc and the top with a strain free glass ring. The stators of which there are a number each having different diameters are also made of stainless steel and embedded in the surface of each is a copper constantan thermocouple. Stator diameters are about 50 mm., their lengths about 10 cm. and the gap widths vary from 0·27 mm. to 0·83 mm. The stator is mounted on a spindle, connection being made by a coned joint so that the different stators can be interchanged to get annular gaps of different sizes. The spindle is held in bearings which have negligible friction and function simply as guides to prevent the spindle moving laterally. To the top end of the spindle are fixed a screw chuck and a small spherical mirror. The chuck grips the lower end of a torsion wire which extends vertically to another identical chuck attached to an adjustable torsion head. The torsion wire is made of beryllium copper. The angular movement of the stator about its vertical axis is observed with the aid of a light beam reflected from the mirrors and focused on a horizontal curved scale of radius 100 cm. From the measured movement of the stator the viscosity coefficient can be found. When double refraction measurements only are required the stator may be clamped so as not to rotate. The drive is produced by an electric motor connected by V-belt to a pulley wheel on the rotor base, the motor being a

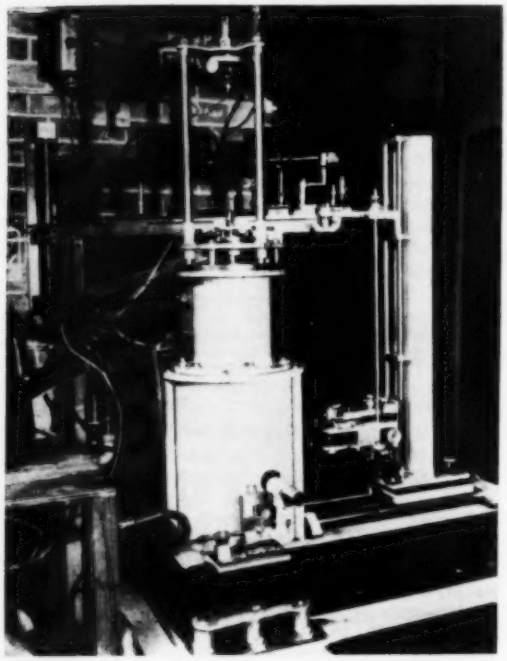

Fig. 3. The apparatus.

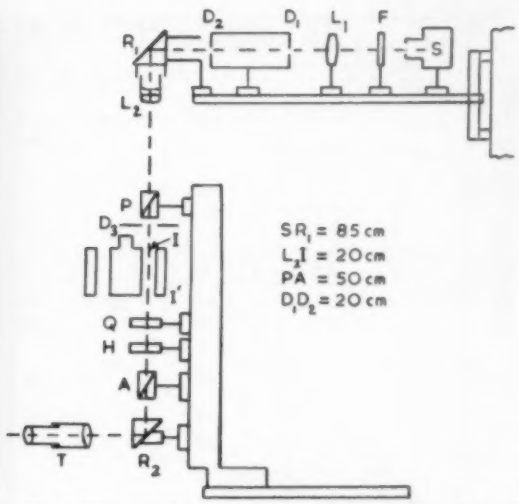

Fig. 4. The optical system. S, light source: F, filter; L_1 and L_2, lenses; D_1, D_2, and D_3, diaphragms; R_1 and R_2, right-angle prisms; P, polarizer; A, analyzer; Q, quarter-wave plate; H, half-shadow (Nakamura) plate; T, telescope; II', cylinder assembly.

variable speed three phase A.C. commutator type with a speed range of 0 to 2,500 r.p.m. With the gap widths available gradients up to 4,000 π sec^{-1} can be produced. The speed is easily adjustable with a smooth and uniform acceleration: both directions of rotation are possible.

The coolant used for temperature control is a mixture of emulsified oil and water: this is used because, if the seals protecting the bearings leak, no harm to the latter can occur. The coolant is pumped continuously from a thermostatted storage tank through the jacket and back to the tank. Temperature is readily controlled to about $\pm$ 0·1°C. which is adequate as it is not claimed that other measurements are so refined as to demand a more stringent control. Measurements were made of the temperature of the liquid in the annulus using the thermocouple embedded in the stator: the rise was always small and an equilibrium temperature quickly reached.

The optical system is shown in Fig. 4: part is mounted on a vertical bench and part on a horizontal bench. This division is purely for convenience of adjustment since one long bench would have been impractical. The light source S is a 250 W. mercury arc giving an intrinsic brightness of 18,000 stilb. The filter F is a Wratten 77A. The lens L_1 has an aperture of 3·5 cm. and a focal length of 10·5 cm.: it produces an image of the arc on a small circular diaphragm D_1. The lens L_2 is an achromat of aperture 1·8 cm. and focal length 14 cm.: it produces an image of D_1 at the cell entrance I and an image of another diaphragm D_2 at H. By this arrangement (Bjornstahl, 1939) unwanted reflections from the cylinder walls were eliminated. P and A are Glan-Thompson polarizing prisms. Q is a quarter-wave plate compensator and H a Nakamura half-shadow plate. Q and H together form an optical compensator which is the unit for measuring the double refraction Δ. The working of this compensator is described elsewhere (Jerrard, 1954). T is a low power telescope (magnification $\times$ 14) focused on the dividing line of the half-shade H.

The whole apparatus including the motor is mounted on a massive concrete base to prevent vibration. Where necessary and possible anti-vibration mountings were used. It is essential to carry out experiments carefully and to eliminate as far as possible sudden jolts and large speed fluctuation since these can easily bring about turbulence at high speeds. The accuracy is such that values of Δn of the order 10^{-8} can be measured to within 2 per cent. The errors in the viscosity coefficient η_i do not exceed 5 per cent: it should be emphasized that highly accurate values of η_i are not necessary in streaming double refraction studies. Values of χ near 45° are considered to be subject to an error of between 3 and 4 per cent: this decreases to about 1 per cent as χ decreases towards zero. The error in the values of G calculated from $G = \omega R/t$, where ω is the angular velocity of the rotor, R its radius and t the gap width, is less than 3 per cent with a movable stator and considerably less than 1 per cent with the stator clamped.

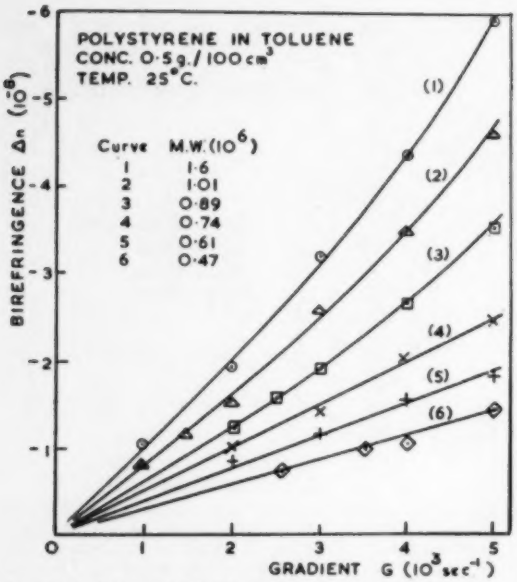

Fig. 5. Variation of birefringence with gradient for polystyrene of different molecular weights in toluene.

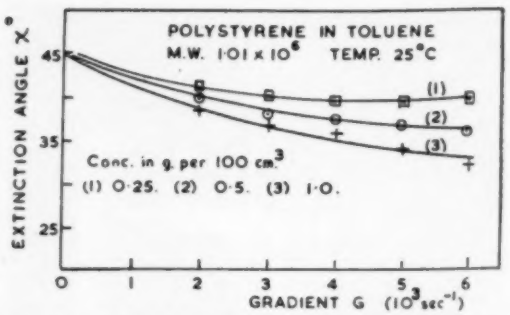

Fig. 6. Variation of extinction angle with gradient for polystyrene in toluene for different concentrations.

Results

Results obtained with the apparatus for polystyrene fractions are shown in Figs. 5 and 6. The polystyrene was provided by the courtesy of B.X. Plastics Ltd. The most concentrated solution of each fraction was made by dissolving a weighed amount of polystyrene in toluene with gentle swirling and diluting to volume at 25°C. Other solutions were prepared by diluting a measured volume of this solution with a measured volume of solvent both at 25°C. From the results it was possible to calculate $(-\delta\chi/\delta G)_{G \to 0}$ for the different concentrations used and from a graph of these values against concentration it was possible to extrapolate to get the value for $(-\delta\chi/\delta G)_{G \to 0}$ for zero concentration. According to theory this latter value is equal to 1/12D so that D can be found. The data for the higher molecular weights is summarized in Table 1. The graphs show that polystyrene of molecular weight less than about $0{\cdot}7 \times 10^6$ exhibit a linear dependence of Δn on G over the gradient range 0 to 5,000 sec^{-1}. The birefringence is negative. The fact that for higher values of molecular weight the rise of Δn with G is more than linear indicates a soft molecule. It was also found that as the molecular weight increases so, for any gradient, χ decreases.

A number of other studies have been made using this apparatus. It appears that in addition to determining rotary diffusion constants the method might be extended to give reliable data in such fields as molecular weight determination and the investigation of polymerization kinetics.

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2·57	1·60	3·6	2,300

$[\eta]$ Intrinsic viscosity.

M Molecular weight calculated from $[\eta] = 1{\cdot}3 \times 10^{-4} M^{0{\cdot}695}$.

$[\chi] = (-\delta\chi/\delta G)_{G \to 0}$

D Diffusion Constant.

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B.S.I.R.A. APPOINTMENT

Mr. R. E. Fischbacher, B.Sc., A.R.C.S.T., A.M.I.E.E. has been appointed an Assistant Director of the British Scientific Instrument Research Association. Educated at the Royal College of Science and Technology and Glasgow University, Mr. Fischbacher served for 12 years in the Admiralty Signal and Radar Establishment as a member of the R.N.S.S. before joining the British Scientific Instrument Research Association in 1957 to become Head of the Electronics Department.

THE OSCILLATING DISC VISCOMETER

R. Roscoe, Ph.D., F.Inst.P.

Senior Lecturer, Physics Department, King's College, Newcastle-upon-Tyne, 1

Summary

The viscometer usually associated with the name of O. E. Meyer is extremely simple to construct and has in the past been used for a number of viscosity measurements, particularly on liquids at low temperatures. Inaccurate results were obtained, partly because incorrect theoretical formulae were used for calculation and partly because of unsuspected sources of experimental error. An adequate theory of the instrument has recently been given by Azpeitia and Newell, and the practical use of this theory (which is applicable to cylinders as well as to discs) is explained in the present paper. The elimination of experimental errors is also discussed, and it is shown by a series of measurements on the viscosity of water that absolute results can be obtained with an accuracy better than 1 per cent.

Introduction

When a rotational motion is imparted to a horizontal disc suspended by a torsion wire, the logarithmic decrement of the subsequent oscillations depends on the viscosity of the surrounding fluid. This gives an apparently simple and accurate method for measuring liquid viscosities of the order of 1 cP which has been extensively employed (particularly for work at very low temperatures where manipulation of the liquid is difficult). For gases it is necessary to have two parallel plates fixed close above and below the disc, but this is an unnecessary complication in the case of liquids which is considered here. Unfortunately, measurements by this method have commonly given results differing by 10 per cent or more from those obtained by capillary tube measurements. This has been in part due to the neglect of an 'edge correction' which must be applied to the original formula for the logarithmic decrement given by Meyer (1861) or to the use of an incorrect edge correction. Failure to take account of a number of other corrections and certain sources of experimental error has also contributed substantially to the errors in the results.

As the type of measurements required and even the design of the apparatus are intimately connected with the formula to be used for the calculation of the results, the following discussion proceeds from a consideration of that formula to the details of design and measurement. It concludes with a series of results obtained with water as a standard liquid which provides an illustration of the accuracy attainable with this instrument.

The Decrement Formula

A rigorous theory of the oscillating disc has only recently been developed by Azpeitia and Newell (1959). Their formulae involve the quantity d, known as the penetration depth of the disturbance in the liquid, defined by

$$d^2 = \frac{T\eta_l}{\pi\rho} \qquad \ldots\ldots (1)$$

where T is the period of oscillations in the liquid, η_l the viscosity of the liquid and ρ its density. When d is much less than the thickness H and radius R of the disc, the theory leads to the 'thick disc' formula:

$$\frac{4I\Delta}{R^4(\pi\rho\eta_l T)^{\frac{1}{2}}} = a_o + 2{\cdot}408\frac{d}{R} + 0{\cdot}944\,a_2\left(\frac{d}{R}\right)^2 + \ldots + \frac{2H}{R}\left\{a_o + \frac{3}{2}\frac{d}{R} + \frac{3}{16}a_2\left(\frac{d}{R}\right)^2 + \ldots\right\} \qquad \ldots\ldots (2)$$

where I is the moment of inertia of the disc and $2\pi\Delta$ is the logarithmic decrement measured between successive swings in the same sense. The quantities a_o and a_2 are given by the series

$$a_o = 1 - \frac{3}{2}\Delta - \frac{3}{8}\Delta^2 \ldots\ldots$$
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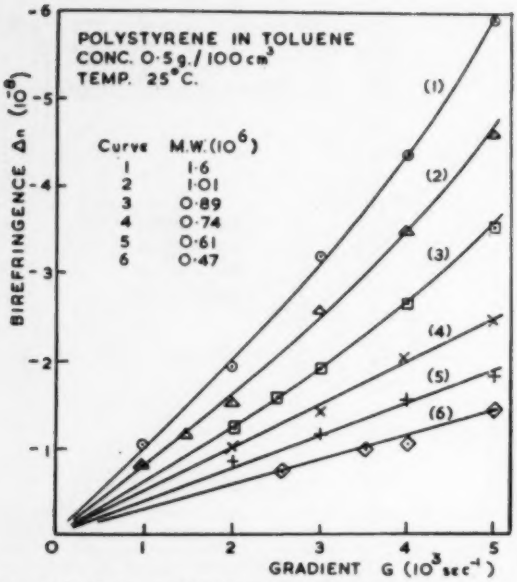
Further terms occur in the series in (2), but as the largest of these are of the order $(d/R)^4$ they are negligible in practical cases (d being of the order 1 mm. and R a few centimetres).

Azpeitia and Newell have also derived a 'thin disc' formula for the case when d is much larger than H (but still less than R) and have given a graphical interpolation between the two. When H is equal to d, their graphs show that the term with the coefficient 2·408 in (2) is effectively reduced by 1 per cent, and the term with the coefficient 0·944 by 0·5 per cent; reductions which generally produce a negligible effect on the result. When H is as low as 0·5 d, the reductions are more serious, being 4 per cent and 1 per cent respectively. In practice, however, it is convenient to make the disc at least 2 mm. thick and this usually implies that H is somewhat greater than d and formula (2) can be used as it stands.

The viscosity η_l appears on the left hand side of (2) explicitly, but it also occurs on the right-hand side through the quantity d. The calculation of η_l has



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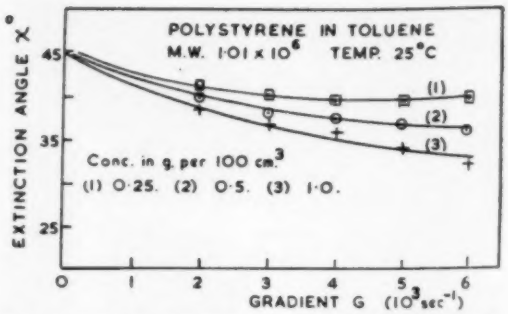

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$$a_2 = 1 + \frac{1}{2}\Delta + \frac{1}{8}\Delta^2 \ldots. \qquad \ldots\ldots (3)$$

Further terms occur in the series in (2), but as the largest of these are of the order $(d/R)^4$ they are negligible in practical cases (d being of the order 1 mm. and R a few centimetres).

Azpeitia and Newell have also derived a 'thin disc' formula for the case when d is much larger than H (but still less than R) and have given a graphical interpolation between the two. When H is equal to d, their graphs show that the term with the coefficient 2·408 in (2) is effectively reduced by 1 per cent, and the term with the coefficient 0·944 by 0·5 per cent; reductions which generally produce a negligible effect on the result. When H is as low as 0·5 d, the reductions are more serious, being 4 per cent and 1 per cent respectively. In practice, however, it is convenient to make the disc at least 2 mm. thick and this usually implies that H is somewhat greater than d and formula (2) can be used as it stands.

The viscosity η_l appears on the left hand side of (2) explicitly, but it also occurs on the right-hand side through the quantity d. The calculation of η_l has

therefore to be made by successive approximations starting with d set equal to zero or some assumed value. As the terms involving d/R are much less than the terms involving a_o the process is rapid and only represents a small part of the total numerical work which has to be performed.

The Suspension Correction

Equation (2) makes no allowance for the imperfect elasticity of the torsion-wire suspension. This can be taken into account by replacing the quantity Δ where it occurs on the left-hand side by

$$\Delta - \lambda \Delta_o$$

where $2\pi\Delta_o$ is the logarithmic decrement when the disc oscillates in vacuo and λ is the ratio of the period of oscillation in the liquid to that in vacuo. The correction can be determined directly by enclosing the apparatus and measuring λ and Δ_o after evacuation. Unfortunately, although the effect of the air on the decrement will drop somewhat during the early stages of evacuation it will then remain at an appreciable value until the pressure is so low (10^{-4} mm.) that the mean free path of the molecules is much less than the disc radius. It is therefore much simpler to obtain Δ_o by measuring the value of Δ when the disc is oscillating in air and subtracting from this the theoretical value of Δ for air oscillations (which is by no means negligible) calculated from (2). As the period in vacuo differs inappreciably from that in air, the value of λ can be obtained using the latter quantity.

It is important to choose a torsion wire of some material such as tungsten which will give a low value of Δ_o which does not vary much with the amplitude of oscillation. Some variation always occurs, so it is necessary that the air decrement observations shall be made over the same range of amplitudes as the decrement measurements in the liquid. It should be noted, however, that the suspension correction can be effectively eliminated by using a good bifilar suspension instead of a torsion wire. Such a suspension produces a negligible contribution to the decrement, but it involves some complication of the apparatus.

The Apparatus

The disc should be fitted with a stem leading up to the suspension and carrying a small plane mirror as shown in Fig. 1. The stem is made thin so that the liquid exerts a negligible drag upon it. This also ensures that the stem contributes a negligible amount to the moment of inertia I of the whole so that, in the absence of other suspended parts beyond the small mirror, the value of I can be calculated as the product of the mass of the disc and half the square of its radius (provided the thickness is uniform). It must be remembered, however, that a variation of 2 per cent in thickness between centre and perimeter can lead to an error of 0·5 per cent in the calculated value of I, and hence to an error of 1 per cent in η. If the disc is made at least 2 mm. thick it is easy to secure the necessary uniformity, and at the same time unnecessary complications in the viscosity calculation are avoided as indicated earlier. The disc radius should be made as large as possible, but the clearance between disc and vessel walls must be at least five times the penetration depth d.

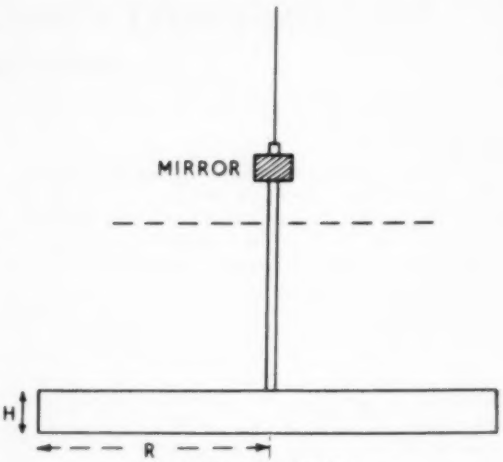

Fig. 1. The oscillating disc.

The disc must hang so that its plane is truly horizontal and hence at right-angles to the axis of rotation. This can be arranged, even if the suspension has not been attached at a point exactly on the disc axis, by a slight bending of the stem. A good torsion head is necessary so that no appreciable horizontal velocity is imparted to the disc when the rotation is started. If the motion of the disc departs visibly from a pure rotation in its own plane, inconsistencies appear in the decrement measurements.

The amplitudes of the disc oscillations are most easily measured by observing the image of a fixed horizontal scale in the mirror through a telescope. The standard arrangement of galvanometer lamp, scale and concave mirror should not be used, as very slight lateral displacements affect the deflection of the beam from a concave mirror; but that system can be modified by using a focusing lens between the lamp and a plane mirror. Ideally, the scale should be curved, but as rather large errors can result from incorrect curvature it is often better to use a straight scale and apply the necessary corrections to the larger amplitude readings.

The Observations

The rotation of the disc is not purely a damped oscillation, as non-oscillatory modes are also present. These die away rapidly, but they produce an asymmetry of the earlier oscillations about the rest position. Their effect can be largely eliminated by using the mean of successive left and right deflections in calculating the decrement. Another difficulty arises from the fact that when the amplitude of oscillation is large, the motion of the liquid is no longer determined by the linear Stokes equations and the decrement rises above the

value given by (2) which is deduced from those equations. Hence it is necessary to check that the decrement is independent of amplitude up to the highest value used. The constancy of the period T over the whole amplitude range must also be checked, particularly if a bifilar suspension is used.

The logarithmic decrement can be obtained graphically from the observed amplitudes of oscillation, but attention must be paid to the low weight of the smaller amplitude observations in drawing the straight line on the graph with logarithmic ordinates. In any case it is difficult to measure the decrement thus to an accuracy of more than 0·5 per cent (giving an accuracy of about 1 per cent in η_t) so that the least-squares method given by Roscoe and Bainbridge (1958) should be used for accurate work.

Due attention must be paid to temperature control and measurement, and to the accuracy of subsidiary observations. In the series of measurements on water shown below, the temperature t was measured to within 0·05 °C., each value of the logarithmic decrement was obtained from the average of three runs with a standard error of 0·2 per cent and the period T was measured to within 0·1 per cent. The errors from other measurements were smaller, and the three listed contribute 0·1, 0·4 and 0·1 per cent respectively to the error in η_t. As these are random errors, the standard deviation of the observed values of η_t arising from experimental inaccuracy should be only just over 0·4 per cent. As equation (2) applies to a long cylinder as well as to a disc, measurements were made with a cylinder and two discs having the following dimensions and mass:

R (cm.)	H (cm.)	Mass (g.)
0·9511	13·483	321·04
3·182	0·2990	79·433
4·448	0·0847	43·164

Two different torsion wires were used for each and the following results obtained:

T (sec.)	Δ x 10^5	$\lambda\Delta_0$ x 10^5	t (°C.)	Obs. η_t (cP)	Std. η_t (cP)	$\frac{H}{d}$
3·152	1,587	58	17·87	1·055	1·057	131
3·129	1,640	58	15·42	1·129	1·125	127
7·66	4,126	61	17·33	1·078	1·072	1·84
17·42	6,322	54	17·33	1·071	1·072	1·23
5·185	9,566	68	15·14	1·139	1·133	0·62
9·22	12,060	31	17·55	1·068	1·066	0·48

In this table the observed results for η_t, computed by means of equation (2), are compared with standard values for the temperature concerned. These were obtained by interpolation between the figures given in Kaye and Laby (1959) which are certainly correct to 0·1 per cent. The agreement is always within 0·6 per cent as might be anticipated from the standard deviation estimated above. It should be noted, however, that the ratio H/d is appreciably less than unity in the case of the thinnest disc (the last two observations in the table), and corrections should here be applied when using equation (2) for the calculations. Azpeitia and Newell's graphs give corrections which would raise these two last values of η_t by 0·5 and 0·7 per cent respectively, giving discrepancies of 1 per cent between observed and standard values; but it may be that the graphical interpolation used by those authors is not very accurate. For this reason, and in order to avoid unnecessary complication of the calculations, the disc thickness should be made at least as great as the penetration depth.

With this viscometer, the great advantage of simplicity of design has to be set off against the length of time taken by the observations and calculations. There is unfortunately no way of simplifying the latter without introducing gross errors into the results, nor is it profitable to calibrate the instrument with liquids of known viscosity. A suitable calibration curve could be constructed, paying due regard to the form of equation (2), but this would involve measurements on a large number of standard liquids of different density and viscosity. The final calculation would be shortened inappreciably and the cumulative errors of the calibration measurements would reduce the accuracy of the result.

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N.E.L. Annual Report 1960

Results of research on machine tools, ball bearings, fatigue at high temperatures, and extrusion of metals are some of the items of direct industrial interest included in the latest Annual Report of the National Engineering Laboratory, which has just been published. Instruments for measuring the air content of liquids and for determining the rate of flow of water in large pipes are other examples of new developments at the Laboratory which will find diverse application in industry.

Background research forms an important part of the Laboratory's work. The report gives details of the first stages of the synthesis of very hard materials (small diamonds were produced recently at N.E.L.); the preparation of new steam tables; the lubrication of gears; and the fracture of metals under combined creep and fatigue.

An account is also given of new research facilities, including a master gear-generating machine, equipment for calibrating gas and oil flowmeters, and rigs for testing the performance of fans. Sponsored work for individual firms has ranged from noise measurement in a factory to efficiency tests on a water turbine.

The report also contains brief notes on all research in progress during the past year.

NOTES ON THE MEASUREMENT OF VISCO-ELASTICITY IN MATERIALS OF HIGH VISCOSITY

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Summary

The Weissenberg-Roberts Rheogoniometer has been redesigned to permit the measurement of the rheological properties of bitumens and similar high viscosity and elastic materials over the range −40 to +350°C.

The modifications include a more powerful drive unit of wide speed range giving platen speeds from one to $10^{-5.9}$ revolutions per second, stiffer construction, and stress measuring devices capable of handling shear stresses to 4×10^6 dynes/cm.² and a total normal stress to 7×10^5 dynes/cm.². Accurate measurement of total normal stress is made possible by a null method employing a simple servo-system.

Techniques have been developed to enable the shear stress for various times of application of the shear strain to be determined. Short times give a peak measurement of stress due to elasticity. Long times of application are useful in evaluating shear stress reduction arising from structural changes and from the heating effect (for which the maximum temperature rise has been calculated).

Cavitation occurs at high rates of shear, making oscillation tests essential, and a new technique devised for measuring phase difference is accurate to ± ½°.

The overall accuracy of measurement of the various rheological parameters is of the order of 5 per cent. This enables differences of comparatively small rheological significance in the behaviour of highly viscous and complex materials to be evaluated.

Introduction

Advances made by various theoreticians[1–27] in the understanding of the fundamental mechanical properties of visco-elastic materials have demanded a corresponding improvement in the experimental testing techniques[28–57].

Weissenberg[5,30] formulated the principles of a testing instrument which he termed the 'rheogoniometer' because, in its ideal form, it would yield a complete specification of rheological data round the full solid angle in space by measuring at every point in the flowing material the development in time of the stresses and strains. The preliminary development of this apparatus has been reported by Russell[29] and Freeman[28] and a modified form for the study of molten plastics has been described by Pollett and Cross[33]. Further substantial improvements were made by Jobling and Roberts[52] and for some years instruments have been available from Farol Research Engineers Limited of Bognor Regis.

The authors are particularly concerned with the study of the rheological characteristics of bitumens over a wide temperature range. The difficulties inherent in measuring the rheological properties of such high viscosity materials have led to further major design changes in the instrument.

The materials investigated show reductions in tangential and normal stresses with time of shear similar to those of plastics. These reductions are due to four effects:

(1) A reversible shear-thinning or 'break-down'.
(2) A form of cavitation or 'break-up'.
(3) The heat effects.
(4) The elastic properties of the liquid which make the peak shear stress dependent on the rate of application of the shear strain.

It is important that the instrument should be able to differentiate between these four effects and determine separately the amount due to each. A technique has been developed to obtain the 'true' viscosity for a given rate of shear, so overcoming the effect of shear-thinning with time of shear. Cavitation makes oscillation tests essential and for this a convenient method of determining phase difference has been devised. To allow for heat effects, the maximum temperature rise has been calculated for a range of viscosities and rates of shear. The rate of application of the shear strain can readily be controlled by altering the clutch engagement time as described elsewhere[52].

Principles of Design

In the design of a rheogoniometer, particular attention must be paid to quality of the bearings, platen alignment and response time of the instrument and its associated equipment.

Basically, the instrument is that developed for visco-elastic liquids and previously described[52]. In addition to the design changes required in measuring high viscosities (more powerful motors, stiffer construction and stress measuring devices of wider range), modifications were also made to improve the three factors mentioned above. The precession of the main journal bearing was limited to 1×10^{-4} in. by careful machining and installation. There is an unavoidable vertical movement of the bearing during rotation of 5×10^{-5} in. However, as a consequence of using a diaphragm drive between the bearing and the platen holder, the vertical movement of the lower platen was found by measurement to be less than 2×10^{-6} in.

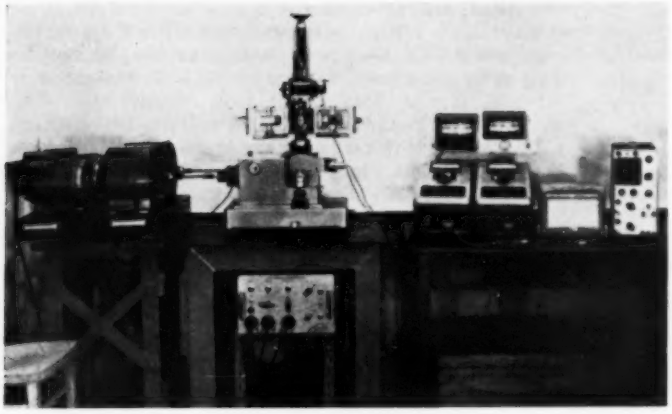

Fig. 1. General layout of Rheogoniometer.

Parallelism of better than 5×10^{-2} degrees between the platens is obtained during rotation and oscillation experiments.

It is essential that the response time of the instrument, including the measuring and recording devices, be less than that of the material under test. The stress measuring devices must consequently respond to small movements and the associated electronic equipment must be of high sensitivity and drift-free.

By the adoption of these refinements, it is believed that the accuracy of measurements has been improved and small differences in the rheological parameters of the materials examined can be distinguished.

Description of the Rheogoniometer

Fig. 1 shows the general layout of the apparatus and Fig. 2 is a close-up of the main frame. Fig. 3 is a labelled diagrammatic sketch of the instrument without the two-way drive box and clutch. The present instrument employs two ½ h.p., 1,500 r.p.m. synchronous motors which drive gear boxes having gear ratios of 1:1 to $10^{-5 \cdot 9}$:1 in 60 steps related logarithmically. The required speed is then transmitted to the platens via an electromagnetic clutch for rotation. Special couplings prevent vibration from the drive unit which is mounted separately from the main frame, which is supported on a vibration-free mounting and housed in a temperature controlled cabinet. Both the drives enter the main frame through the two-way drive box and turn the bottom platen (A) through the worm wheel (B). The bottom platen is rotated by turning the worm (C) continuously, whilst oscillatory motion results from back and forth axial movement of the worm. As the instrument itself incorporates a speed reduction of 25:1, the lower platen speeds obtained range from one to $10^{-5 \cdot 9}$ revolutions per second. Hence the range of rates of shear available, depending upon the cone angle, are from about 10^{-4} to 10^{3} sec^{-1}. For oscillation, the frequency range is 25 cycles per second to $2 \cdot 5 \times 10^{-5}$ cycles per second. The amplitude of oscillation of the

Fig. 2. Close-up of instrument.

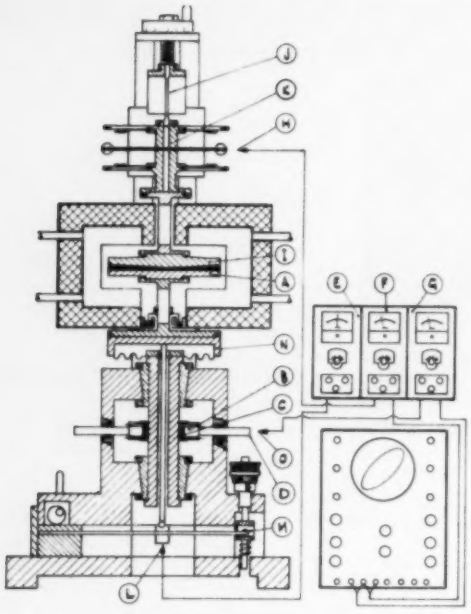

Fig. 3. Diagram of instrument

worm shaft (D) is variable from 1 to 50 $\times$ 10^{-3} in. which is equivalent to a movement of the lower platen from ·0006 to 0·03 radians.

The stress measuring devices previously used employed condenser gauges. These have been replaced by selected and modified inductive displacement transducers used in conjunction with 'Pendeford Multimeters' (E. F. G) manufactured by Boulton Paul Aircraft Ltd. These transducers have two advantages over condenser gauges; firstly, the sensitivity is unaltered by small movements, and secondly, they require only one adjustment for zero setting. The d.c. signal produced by the Multimeter, can be used to operate an oscillograph or recording equipment. The minimum full-scale deflection which can be measured, using these specially selected transducers, is 62·5 micro-inches to an accuracy of 1/100 of this. A transducer (H) measures the total torque on the upper platen (I) in conjunction with a torsion bar (J) clamped in the torsion head (K). The torsion head is more complex than that previously used, and is capable of withstanding large stress. The rigidity of construction allows only negligible vertical movement. The upper platen is restrained from sideways movement by the two sets of three wires in the torsion head. Another transducer (L) measures the thrust on the lower platen, i.e. the total normal force, against the spring (M). The lower platen assembly (N) may be either of the 'solid' type (no normal force measurement) or 'flexible' for normal force measurement. The standard 'flexible' lower platen assembly has also been modified so that it can be locked solid so that materials are more easily loaded into the gap. In order to cover a wide range of forces, the torsion bar and spring can be replaced by others of different 'stiffness' coefficients and the diameter of the three wires in the torsion head can be chosen to change the capacity of the total torque measurement. Table I gives the ranges of various combinations.

A transducer (O) indirectly also measures the amplitude of oscillation and phase of the strains given to the material under test.

The gap between the platens is accurately set by a transducer mounted vertically on the torsion head.

The platens are so designed that they are readily interchangeable. Thus, combinations of diameters of cones of various angles and plates, up to a maximum of 7·5 cm., are used in either the top or bottom positions.

Recording Apparatus.—The d.c. signals from the Multimeters can be used to drive other items of equipment.

1. For rapid changes this output may be recorded on the screen of a cathode ray oscillograph and photographed.
2. For slow phenomena, the signals can drive a direct inking recorder or 'X-Y' co-ordinate plotting table.

Temperature Control and Measurement.—For temperature control the platens are surrounded by an openable, insulated, hollow box through which is circulated liquid at a constant temperature. Kerosine is used for temperatures from −40 to +60°C., and a special mineral oil from 40 to 150°C. The platens are mounted on insulated shafts and a liquid seal fits around the shaft of the lower platen. For work on molten polymers up to a temperature of 350°C., an electric oven is used.

Each of the interchangeable upper platens is fitted with an iron/constantan thermocouple with its junction in the surface of the platen. The thermocouple wires are taken up the shaft and attached to a reference junction which is located within the temperature controlled chamber. A sensitive galvanometer measures any change in the temperature of the material near to the platen. In fact the thermocouple cannot measure the actual change in the temperature of the material because this would require location of the thermocouple junction in the material itself, so distorting the flow conditions.

An important factor to be taken into account is the dissipation of energy during flow. If heat losses are

TABLE 1
Range of Stress Measurements

(1) *Tangential Stress, dynes/cm.*^{2}

Head	*Light* (0·020 *in. diam. wires*)			*Heavy* (1/16 *in. diam. wires*)		
Torque Bar diam., in.	0·1	0·2	0·3	0·1	0·2	0·3
2·5 *cm. diam. Platen*						
Minimum	$2{\cdot}144 \times 10$	$8{\cdot}633 \times 10$	$4{\cdot}197 \times 10^2$	$1{\cdot}007 \times 10^2$	$1{\cdot}799 \times 10^2$	$4{\cdot}796 \times 10^2$
Maximum	$1{\cdot}487 \times 10^5$	$8{\cdot}250 \times 10^5$	$3{\cdot}070 \times 10^6$	$7{\cdot}578 \times 10^5$	$1{\cdot}439 \times 10^6$	$3{\cdot}645 \times 10^6$
7·5 *Diam. Platen*						
Minimum	$7{\cdot}941 \times 10^{-1}$	$3{\cdot}198$	$1{\cdot}554 \times 10$	$3{\cdot}731$	$6{\cdot}662$	$1{\cdot}776 \times 10$
Maximum	$5{\cdot}507 \times 10^3$	$3{\cdot}055 \times 10^4$	$1{\cdot}137 \times 10^5$	$2{\cdot}807 \times 10^4$	$5{\cdot}329 \times 10^4$	$1{\cdot}350 \times 10^5$

Above table calculated using the following deflections of the torque bar at 7 cm. from its centre:
Max. deflection $= 1 \times 10^{-2}$ in.
Min. (2 per cent of Multimeter full-scale deflection of 62·5 micro-inches) $= 1.25 \times 10^{-6}$ in.

(2) *Total Normal Stress, dynes/cm.*^{2}
(*Measured using servo-system*)

Platen diam., cm.	2·5			7·5		
Spring Thickness, S.W.G.	20 (0·036 in.)	18 (0·048 in.)	16 (0·064 in.)	20 (0·036 in.)	18 (0·048 in.)	16 (0·064 in.)
Minimum	$1{\cdot}300 \times 10$	$3{\cdot}147 \times 10$	$7{\cdot}274 \times 10$	$1{\cdot}443$	$3{\cdot}497$	$8{\cdot}083$
Maximum	$9{\cdot}606 \times 10^4$	$2{\cdot}598 \times 10^5$	$7{\cdot}095 \times 10^5$	$1{\cdot}067 \times 10^4$	$2{\cdot}887 \times 10^4$	$6{\cdot}262 \times 10^4$

Above table calculated from the following deflections measured at the end of the spring:
Max. deflection $= 1 \times 10^{-1}$ in.
Min. (2 per cent of Multimeter full-scale deflection of 1×10^{-3} in.) $= 2 \times 10^{-5}$ in.

Note: The diameter of the platens could be increased to a maximum of 15 cm.

neglected, the maximum temperature rise per second can be calculated from the platen geometry, the viscosity, specific heat of the material and the rate of shear. The results are given for a typical bitumen in Fig. 4 for rates of shear from 0·01 to 100 sec^{-1} for viscosities from 10^2 to 10^8 poise. At low rates of shear and at low viscosities it will be evident that these temperature rises can be neglected. However, it also follows that, according to the viscosity and type of bitumen being tested there is a specific rate of shear at which the temperature effect will become greater than that part of the reduction in tangential stress which is due to breakdown of the material with time of shearing.

Normal Force Measurement

The total normal force cannot be measured without some vertical movement of the platens. However, this necessarily introduces inaccuracies in measurement; one is an apparent reduction in the shear stress, and the other a masking of those physical properties which vary with time of shear.

If the lower platen is free to move vertically (the material under test is regarded as incompressible under the small compressive stresses involved) a positive normal stress which produces a movement of the lower platen will have two effects upon the shear stress. The first is that the effective radius of the sample is decreased,

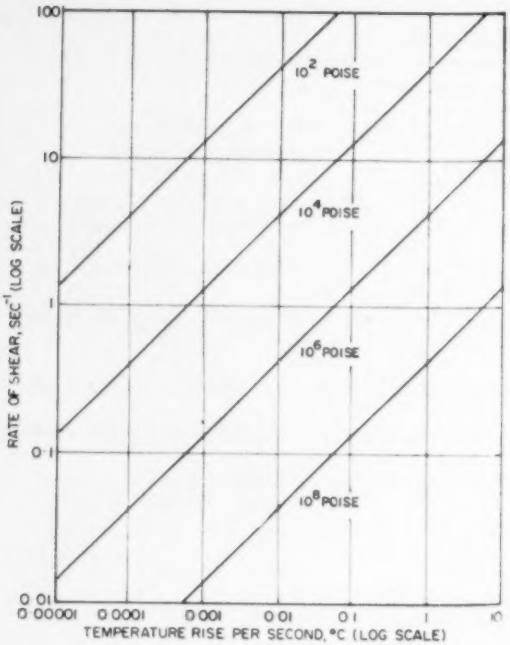

Fig. 4. Temperature rise *v.* rate of shear for viscosities from 10^{-2}–10^{-8} poise.

so the total torque developed will be smaller than that originally applied. The second effect is that the rate of shear is diminished to an extent depending upon the inverse of the radius of the material within the gap, which again reduces the indicated torque. Vertical movement of the platens will also affect the measurement of the normal stress in a similar manner. These effects can be calculated and Table II gives the values for the maximum permissible vertical movement to keep errors of measurement to less than 2 per cent. These values take no account of any deformation of the main instrument structure and connecting members. Calculations show that such deformations will lead to an increase in gap size of about one micro-inch per 100 g. of total applied normal load.

Vertical platen movement is governed by the viscous flow of the material, by the size and angle of the platens and by the spring rate of the normal stress measuring device, and this will affect the time for the normal stress to reach equilibrium much more than for the tangential stress. To improve matters substantially, a sensitive null-point servo-system has been developed so that movements between the platens is less than two micro-inches.

Description of the Servo-System.—A diagrammatic sketch of the servo-system is shown in Fig. 5. A 330

TABLE II

Movements of Lower Platen for 2 per cent error of Tangential and Total Normal Stresses

Cone angle, Degrees	*Platen dia., cm.*	*Movement, in.*
4	2·5	3×10^{-4}
1	2·5	6×10^{-5}
4	7·5	7×10^{-4}
1	7·5	2×10^{-4}

Note: The above table is based on the calculation of the effect of the decrease in radius of the material upon the rate of shear and the subsequent reduction of both the tangential and normal stress.

r.p.m. servo-motor (A) drives the micrometer (B) through a 30:1 gear box (C) and bellows (D). The transducer (E), originally designed for measuring the lower platen movement, now provides an error signal to the Multimeter (F). The output from this multimeter supplies the amplifier (G) which drives the servo-motor, thus raising the end of the spring and keeping the lower platen in a fixed position to within 2 micro-inches. A transducer (H) and Multimeter (I) measure the movement of the end of the spring and the output from the Multimeter can be used for recording the normal stress in the usual manner.

Fig. 6 illustrates the effect of employing the servo-system upon both the tangential and normal stresses.

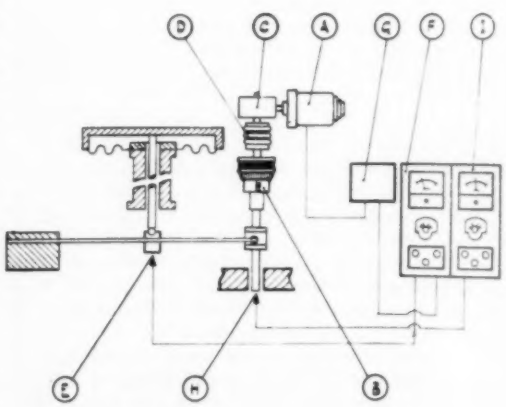

Fig. 5. Normal force servo-system.

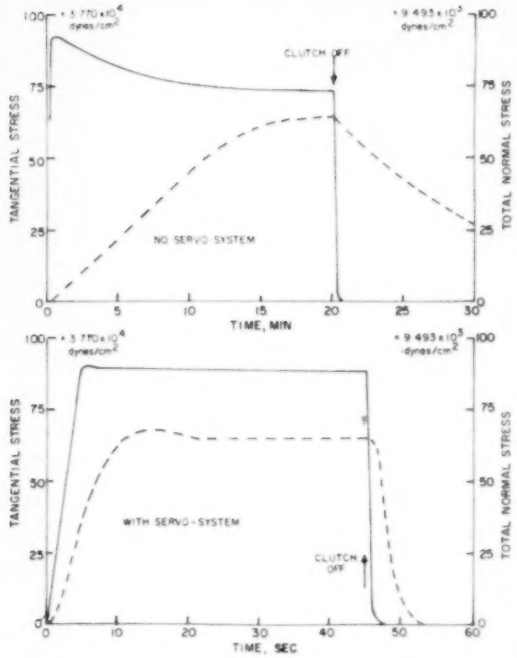

Fig. 6. Effect of normal force servo-system on tangential and total normal stresses. Tangential stress ——— Total normal stress - - - - - - - - - - Rate of shear: 8·545 × 10^{-2} sec^{-1}.

Techniques

Certain techniques of working with such highly viscous and complex materials as bitumens may be of interest.

Filling.—The gap between the platens must first be set with the instrument empty and at the test temperature. The space between the platens is then opened and the required amount of material calculated just to overfill the volume between the cone and plate is placed on the lower platen. The top platen is then lowered to the required gap distance (as indicated by the transducer and allowing for the weight of the material) while the lower platen is rotating slowly. With the most viscous materials heat is also applied. The locking device incorporated in the lower platen assembly protects the normal force measuring system against overload. Time is then allowed for the platens and the material to regain the test temperature; the platen thermocouple is useful in deciding when this temperature is reached. However, with the materials tested, relaxation times have been up to many hours. This necessitates a prolonged wait before testing can be commenced because it is essential that the material is left for sufficient time for it to reach stress equilibrium. Consequently a jig is being designed so that sets of platens can be prepared with the material enclosed at the correct gap setting and then the whole assembly can be fitted into the instrument. Testing can thus take place immediately the material and the platens attain the test temperature.

Experiments at the lower rates of shear and at the lower oscillation frequencies are conducted before those at higher speeds as the least disturbance demands the shortest waiting periods.

Rotation.—The change in tangential stress and in total normal stress with time of shear for various rates of shear are illustrated in Fig. 7. Careful considerations of these various curves has shown that:

1. At low rates of shear there is no change in structure.
2. At higher rates of shear there is a viscosity breakdown to a definite level.
3. At the highest rates of shear the flow is distorted (i.e. cavitation) and the material becomes anisotropic.

'Break-up' of the material is indicated by visible cavitation at the free boundary, i.e. at the edge of the platens. Hence to obtain a true tangential stress and time of shear curve for a rate of shear at which break-up occurs it is necessary to refill the gap for every test at a high rate of shear. However, a special 'overflow' cone and plate enables rotation experiments to be extended to higher rates of shear, and permits the material to heal at the free boundary.

Generally it is the stress at equilibrium which has been used to calculate the apparent shear viscosity (η_s) and hence the idealized shear viscosity coefficient (η_s max.). Unfortunately, this equilibrium stress concept is only valid for those rates of shear where an equilibrium stress is attained, and as has been shown above, this is only true at low or moderate rates of shear. Thus, the peak stress is the only remaining significant parameter common to all curves. However, the peak stress of any particular tangential stress and time of shear curve is not necessarily the value which should be used to calculate the apparent viscosity—it is the *maximum* attainable peak stress, and this is only obtainable if the material has completely relaxed in all planes, that is, when both the tangential and the normal stress are completely relaxed. This maximum peak stress is measured by allowing the material to rest for increasing periods until testing shows that the peak stress has reached a limiting value. These maximum peak stresses can only be measured reliably if the stress measuring device has a very rapid response.

Oscillation.—Oscillation testing, in contrast to simple rotational testing, not only enables measurements to be made of the complex modulus of elasticity (G) but also of the dynamic modulus of viscosity (η_d). These allow comparison with the results of the rotation tests,

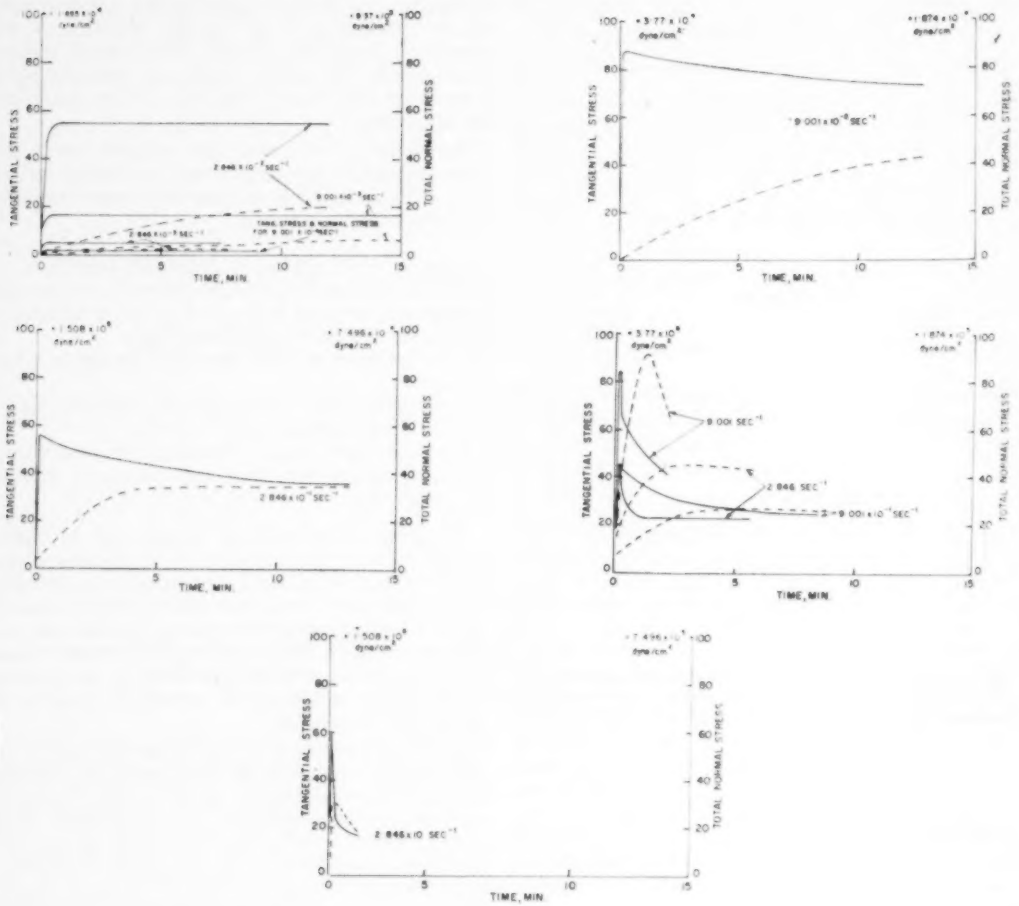

Fig. 7. Tangential stress and total normal stress *v.* time of shear for various rates of shear from 9·001 × 10⁻⁴ to 2·846 × 10 sec⁻¹)
Tangential stress ——— Total normal stress - - - - - - - -

and also permit use of energy inputs at which the material would 'break up' in rotation. At any given frequency the experimentally determined quantities are the amplitude ratio of the applied oscillatory strain and its resultant stress, and the phase difference between these two parameters. In earlier work, the sine waves from the strain and stress were displayed separately on a C.R.O. and photographed, but analysis of the results was tedious and measurement of phase difference was only accurate to ± 3°. Recently, a technique for measurement of phase difference accurate to ± ½° has been devised. The two sine waves representing the strain and stress are displayed at right angles either on an 'x-y' co-ordinate plotting table for frequencies up to 0·25 cycles per second, or on a C.R.O. for frequencies up to the maximum, i.e. 25 cycles per second. The built-in amplifiers of these instruments are used to make the stress amplitude equal to the imposed strain amplitude without introducing phase changes. For zero phase difference the result is a straight line at

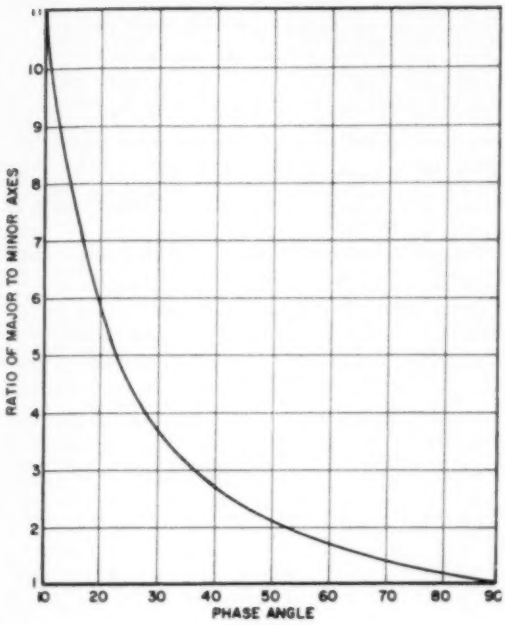

Fig. 8. Ratio of major and minor axes for phase differences of 10° to 90°.

45° to the X axis, and for 90° phase difference the result is a circle. For intermediate phase differences an ellipse results, and the ratio of the major to minor axes of this ellipse is a measure of the phase angle between the two sine waves. Fig. 8 shows the ratio of major and minor axes for phase differences from 10 to 90°. The ellipses in Fig. 9 illustrate for a bitumen the change in phase angle which occurs with increased frequency.

Conclusions

A rheogoniometer has been designed and techniques developed which enable the rheological properties of bitumens and similar high viscosity and elastic materials to be studied over the range from −40 to +350°C.

Shear stresses can be measured for various times of application of shear strain. Long times are useful in evaluating shear break-down effects due to structural changes. Short time of application gives a peak measurement of stress due to elasticity. This peak stress has a particular significance when studying the rheological behaviour of bitumens under the transient loadings encountered in road service.

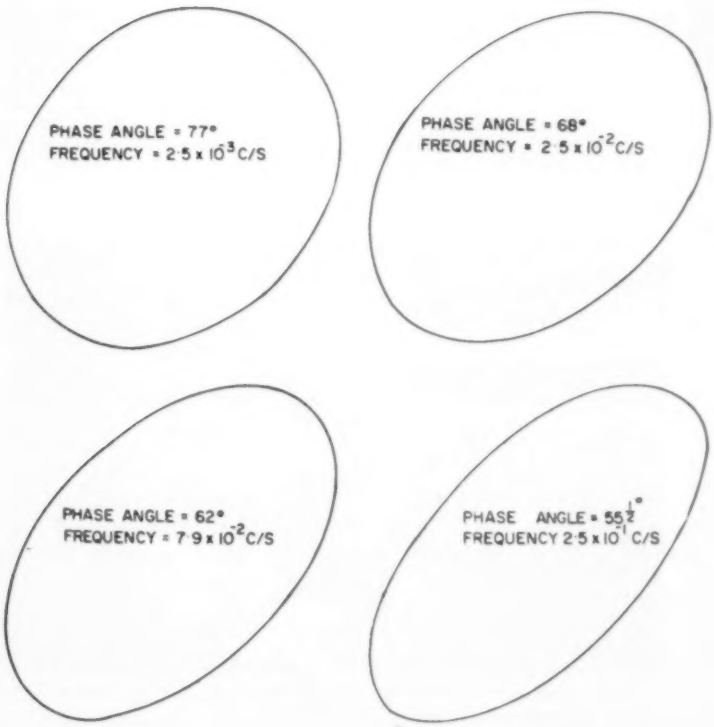
PHASE ANGLE = 77°
FREQUENCY = 2·5 x 10³ C/S
PHASE ANGLE = 68°
FREQUENCY = 2·5 x 10⁻² C/S
PHASE ANGLE = 62°
FREQUENCY = 7·9 x 10⁻² C/S
PHASE ANGLE = 55½°
FREQUENCY 2·5 x 10⁻¹ C/S

Fig. 9. Variation of phase angle with frequency. Liquid = 90° (circle); Solid = 0° (straight line).

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NEWS

New Plessey Laboratory

The new laboratory for the testing and development of aircraft generation systems by The Plessey Company Limited is now operational, and work is expected to step up to full load over the next year. Situated at Eastern Avenue West, Romford, Essex, the plant has been 'custom built' to the specification of the Plessey Works Engineers Department and the laboratory staff themselves, and covers a floor area of 15,000 sq. ft.

The initial installation, currently proving a four generator system of 160 kVA total for the Vickers VC 10, is capable of handling four paralleled channels of equipment up to 120 kVA. normal load per channel with complete simulation of environmental conditions likely to be met by aircraft of the next decade. Future projects include systems testing for the BAC 111 and for military aircraft. The generator drives have a speed range in excess of 3·1 with controlled acceleration up to 1,200 r.p.m. per second and a capacity of 200 h.p. per drive. Three phase loading, both resistive and reactive, is variable between zero and 150 kVA per channel. The comprehensive cooling facilities installed enable direct simulation of supersonic flight conditions.

A hot and cold chamber enables small associated equipments to be checked over the entire environmental temperature range. Temperatures obtainable range from —60°C. to + 120°C., and this range can be totally encompassed within one hour.

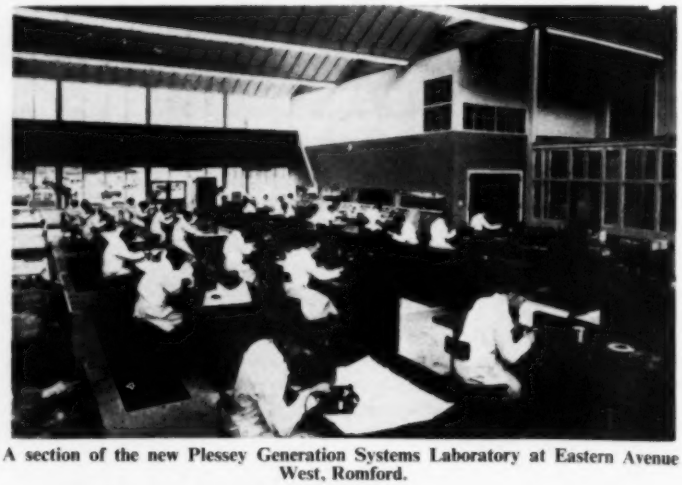
Change of Address

Cawkell Research & Electronics Ltd., have now moved to 99 Uxbridge Road, Ealing, London. W.5. *Tel.:* Ealing 6215 and 7585.

Change of Address

Technicon Instruments Co. Ltd., are now installed in their new laboratories, offices and works at Hanworth Lane, Chertsey, Surrey. *Tel.:* Chertsey 2291-3.

Technicon will hold at Chertsey their regular courses of a week's duration for the instruction of new users of the Autoanalyzer system. These will be conducted, under the guidance of the managing director, Mr. L. J. Evans, by Dr. H. W. Holy and Dr. Martin, Technicon's technical experts.



A section of the new Plessey Generation Systems Laboratory at Eastern Avenue West, Romford.

MEETINGS FOR THE MONTH

Mid-November to Mid-December

We give below a selection of meetings of interest to laboratory workers. Details are as full as possible at the time of going to press, but readers requiring further information or confirmation of arrangements should contact the organizers.

November 8

The Chemical Society (Edinburgh). Joint meeting with the Royal Institute of Chemistry and the Society of Chemical Industry. At the Heriot-Watt College, Edinburgh. 7.30 p.m. 'Some Principles and Practices in Corrosion Protection', by Dr. T. P. Hoar, M.A.

November 9

The Chemical Society (Aberdeen). Joint meeting with the Royal Institute of Chemistry and the Society of Chemical Industry. At Marischal College, Aberdeen. 8 p.m. 'The Discovery of New Drugs', by Dr. A. F. Crowther, M.A.

The Chemical Society (Aberystwyth). Joint meeting with the University College of Wales Chemical Society. In the Edward Davies Chemical Laboratories. 5 p.m. 'The Anatomy of the Chemist', by Dr. T. S. Stevens, A.R.I.C.

The Society of Instrument Technology (Liverpool Section). At the Merseyside and North Wales Electricity Board Industrial Centre, Paradise Street, Liverpool. 7 p.m. 'Our Technology and our Society', by G. C. Eltenton, B.A., F.Inst.P., F.Inst.Pet. Immediate Past President.

November 10

The Chemical Society (Birmingham). Joint meeting with the University Chemical Society. In the Chemistry Department, The University. 4.30 p.m. 'The Active Centres of Enzymes', by Professor H. N. Rydon, D.Sc., F.R.I.C.

The Chemical Society (St. Andrews and Dundee). Joint meeting with the University Chemical Society. In the Chemistry Department, The University, St. Andrews. 5.15 p.m. 'Chemical Relationships between Brewing Beer and Baking Bread', by Dr. M. A. Pyke.

The Chemical Society (Tees-side). Joint meeting with the Royal Institute of Chemistry and the Society of Chemical Industry. At the William Newton School, Norton. 8 p.m. 'Aspects of Chemistry of Olefin Complexes of Transitional Metals', by Professor G. Wilkinson, Ph.D., F.R.I.C.

November 13

The Chemical Society (Cambridge). In the University Chemical Laboratory. 5 p.m. 'The Physical Chemistry of Ion-exchange Polymers', by Dr. E. Glueckauf.

The Chemical Society (Durham). Joint meeting with the Durham Colleges Chemical Society. In the Science Laboratories, The University. 5 p.m. 'Surface Radiochemistry', by Dr. S. J. Thompson, B.Sc.

The Chemical Society (Oxford). Joint meeting with the Alembic Club. In the Inorganic Chemistry Lecture Theatre. 8.15 p.m. 'Some Aspects of the Chemical Structure of Proteins', by Professor H. D. Springall, M.A., D.Phil., F.R.I.C.

The Chemical Society (Swansea). Joint meeting with the University College Chemical Society. In the Department of Chemistry, University College. 4.30 p.m. 'Recent Advances in Nuclear Resonance', by Dr. R. E. Richards, M.A., F.R.S.

The Society of Instrument Technology (Manchester Section). Joint meeting with The Institute of Physics and the Physical Society. At Manchester University. 6.45 p.m. 'The Role of the Physicist in Plant Instrumentation', by L. J. R. Postle, Ph.D., A.Inst.P.

November 14

The Chemical Society (Aberystwyth). Joint meeting with the Royal Institute of Chemistry. In the Edward Davies Chemical Laboratories, Aberystwyth. 5 p.m. 'Nuclear Magnetic Resonance Spectroscopy', by Dr. R. E. Richards, M.A., F.R.S.

The Chemical Society (Manchester). In Room F1, Manchester College of Science and Technology. 6.30 p.m. Centenary Lecture, 'Some Reactions of Free Radicals', by Professor G. B. Kistiakowski.

The Chemical Society (Nottingham). Joint meeting with the University Chemical Society. In the Department of Chemistry. The University. 5 p.m. 'Experimental Methods in the Study of Chemical Kinetics', by Professor J. C. Robb, Ph.D., F.R.I.C.

November 15

The Society for Analytical Chemistry (Scottish Section). At the Royal Society of Edinburgh, George Street, Edinburgh. 7.15 p.m. Meeting on 'Recent Developments in Chromatography on Cellulose and Ion-exchange Cellulose', by N. F. Kember.

The Society for Analytical Chemistry (Midlands Section). At the Technical College, Coventry. 7 p.m. Meeting on 'The Analysis of Finishes on Yarns and Fabrics', by E. Mytum.

The Society of Instrument Technology (Bristol Section). At the Department of Physics, University of Bristol. The Royal Fort, Bristol 8. 7.30 p.m. 'Instrumentation at Low Temperatures', by R. G. Chambers, M.A., Ph.D.

November 16

The Chemical Society (London). In the Rooms of the Society, Burlington House, London, W.1. 7.30 p.m. Centenary Lecture, 'Some Reactions of Free Radicals', by Professor G. B. Kistiakowski.

The Chemical Society (Hull). In the Department of Chemistry, The University. 5 p.m. 'Some Studies in the Porphyrin Field', by Professor G. W. Kenner, Ph.D., Sc.D.

The Chemical Society (Manchester). Joint meeting with the Students Union Chemical Society of Royal Technical College, Salford. In the Conference Hall of the College. 5 p.m. 'The Biogenesis of Alkaloids', by Sir Robert Robinson, O.M., D.Sc., F.R.S.

The Chemical Society (Sheffield). Joint meeting with the Royal Institute of Chemistry and the University Chemical Society. In the Department of Chemistry, The University. 4.30 p.m. 'Polynuclear Complex Formation in Solution', by Dr. F. J. C. Rossotti, M.A.

The Society of Instrument Technology, Data Processing Section. At Manson House, 26 Portland Place, London, W.1. At 6.30 p.m. for 7 p.m. 'Digital Techniques for the Study of Sea Waves, Ship Motion and Similar Processes', by D. E. Cartwright, B.Sc., M. J. Tucker, B.Sc., F.Inst.P., and Miss D. B. Catton, B.Sc.

The Society of Instrument Technology (East Midland Section). At the College of Further Education, Greenclose Lane, Loughborough. 6.45 p.m. for 7.30 p.m. 'A Survey of the International Temperature Scale', by J. A. Hall, B.Sc., A.R.C.S., D.I.C., F.Inst.P.

The Society of Instrument Technology (Grangemouth Section). At the Leapark Hotel, Bo'ness Road, Grangemouth. 7 p.m. 'Development of a Precision Pressure Generator for Multiple Instrument Calibration', by B. E. Adams, B.Sc.,(Eng.), A.M.I.Mech.E.

The Society of Instrument Technology (Newcastle Section). In the Conference Room, Roadway House, Oxford Street, Newcastle-upon-Tyne. 7 p.m. 'Data Handling and Processing', by R. J. McQuaker.

November 17

The Chemical Society (Cambridge). Joint meeting with the University

Chemical Society. In the University Chemical Laboratory. 8.30 p.m. Official Meeting and Centenary Lecture, 'Some Reactions of Free Radicals', by Professor G. B. Kistiakowski.

The Chemical Society (Exeter). Joint meeting with the University Chemical Society. In the Washington Singer Laboratories, Exeter. 5.15 p.m. 'Recent Developments in Acetylene-Allene Chemistry', by Professor E. R. H. Jones, D.Sc., F.R.S.

The Chemical Society (Glasgow). Joint meeting with the Alchemists' Club. In the Chemistry Department, The University, Glasgow. 4 p.m. 'Some Aspects of Cholesterol Biosynthesis', by Dr. G. J. Popjak, F.R.S.

The Chemical Society (Southampton). Joint meeting with the University Chemical Society. In the Chemistry Department, The University. 5 p.m. 'The Rapid Reactions of Some Complex Ions', by Dr. R. G. Wilkins.

The Society of Instrument Technology (Scottish Section). At the Scottish Building Centre, Sauchiehall Street, Glasgow. 7.15 p.m. 'Development of a Precision Pressure Generator for Multiple Instrument Calibration', by B. E. Adams.

November 20

The Chemical Society (Cambridge). In the University Chemical Laboratory. 5 p.m. 'Persulphate Oxidation of Carboxylic Acids', by Dr. R. H. Thomson, F.R.I.C.

The Chemical Society (Leicester). Joint meeting with the University Chemical Society. In the University. 4.30 p.m. 'Some Applications of Electron-spin Resonance Spectroscopy', by Professor H. C. Longuet-Higgins, M.A., D.Phil., F.R.S.

The Chemical Society (Newcastle upon Tyne). In the Chemistry Department, King's College. 5.30 p.m. 'Free Radicals in Irradiated Crystals', by Dr. D. H. Whiffen, M.A.

November 21

The Chemical Society (Keele). Joint meeting with the University College Science Society. In the Department of Chemistry, University College of North Staffordshire. 8.30 p.m. 'The Anatomy of the Chemist', by Dr. T. S. Stevens, A.R.I.C.

The Society for Analytical Chemistry. In the Meeting Room of the Chemical Society, Burlington House, London, W.1. 6.30 p.m. Annual General Meeting followed by the Retiring Chairman's Address.

November 22

The Chemical Society (Dublin). In the Department of Chemistry, Trinity College. 5.30 p.m. 'The Stereochemistry of Flavan-4-ols', by Professor Eva M. Philbin, D.Sc.

November 23

The Chemical Society (Liverpool). Joint meeting with the Student Chemical Society In the Department of Inorganic and Physical Chemistry, The University. 5 p.m. 'Some Aspects of Structure and Reactivity in Ionic Solutions', by Professor K. W. Sykes, M.A., D.Phil.

The Chemical Society (Northern Ireland). Joint meeting with the Royal Institute of Chemistry and the Society of Chemical Industry. In the Chemistry Department, David Keir Building, Queen's University, Belfast. 7.45 p.m. 'Some Aspects of Tannin Chemistry', by Professor R. D. Haworth, D.Sc., F.R.S.

The Society of Instrument Technology (Chester Section) At Stanley Palace, Watergate Street, Chester. 7 p.m. 'Transistors Applied to Modern Instrumentation', by J. E. Fielden, M.Sc., M.I.E.E.

November 24

The Chemical Society (Bristol). Joint meeting with the Royal Institute of Chemistry and the Society of Chemical Industry. At Long Ashton Research Station. Annual Dinner and Dance.

The Chemical Society (Dublin). Joint meeting with the Werner Society. In the Department of Chemistry, Trinity College. 5.30 p.m. 'Some New Photochemical Reactions', by Professor D. H. R. Barton, D.Sc., F.R.S.

The Chemical Society (St. Andrews and Dundee). Joint meeting with the University Chemical Society. In the Chemistry Department, The University, St. Andrews. 5.15 p.m. 'New Reactions in Dinitrogen Tetroxide', by Professor C. C. Addison, Ph.D., F.R.I.C.

November 27

The Chemical Society (Durham). Joint meeting with the Durham Colleges Chemical Society. In the Science Laboratories, The University. 5 p.m. 'Metals and β-Diketones', by Dr. M. R. Truter, B.Sc.

The Chemical Society (Oxford). Joint meeting with the Alembic Club. In the Inorganic Chemistry Lecture Theatre. 8.15 p.m. 'Some Hydrido-complexes of Transition Metals', by Dr. J. Chatt, F.R.S.

November 28

The Chemical Society (Edinburgh). Joint meeting with the University Chemical Society. In the Department of Chemistry, The University. 4.30 p.m. 'Hydrogen Bonding from a Crystallographer's Viewpoint', by Dr. J. C. Speakman, M.Sc.

The Chemical Society (Tees-side). At the Constantine Technical College, Middlesbrough. Official Meeting and Lecture. 'Means to Some Ends', by Professor C. L. Wilson, D.Sc., F.R.I.C.

The Society of Instrument Technology (London). At Marson House, 26 Portland Place, W.1. 6.30 p.m. for 7 p.m. 'Automatic Plant Analysis by Electrochemical Methods', by R. F. Rodger.

November 29

The Society of Instrument Technology (South Wales Section). At the Welsh College of Advanced Technology, Cathays Park, Cardiff. 6.45 p.m. 'Differential Producers for Flow Measurement', by H. E. Dall.

November 30

The Chemical Society (Leeds). In the Chemistry Lecture Theatre, The University. 6.30 p.m. 'Aspects of the Biosynthesis of Phenolic Compounds', by Professor C. H. Hassall, M.Sc.

The Chemical Society (Sheffield). Joint meeting with the Royal Institute of Chemistry and the University Chemical Society. In the Department of Chemistry, The University. 4.30 p.m. 'The Electronic Structures of Some Molecules and Radicals', by Dr. J. W. Linnett, M.A., F.R.S.

December 1

The Chemical Society (Birmingham). Joint meeting with the University Chemical Society. In the Chemistry Department, The University. 4.30 p.m. 'New Reactions in Dinitrogen Tetroxide', by Professor C. C. Addison, D.Sc., F.R.I.C.

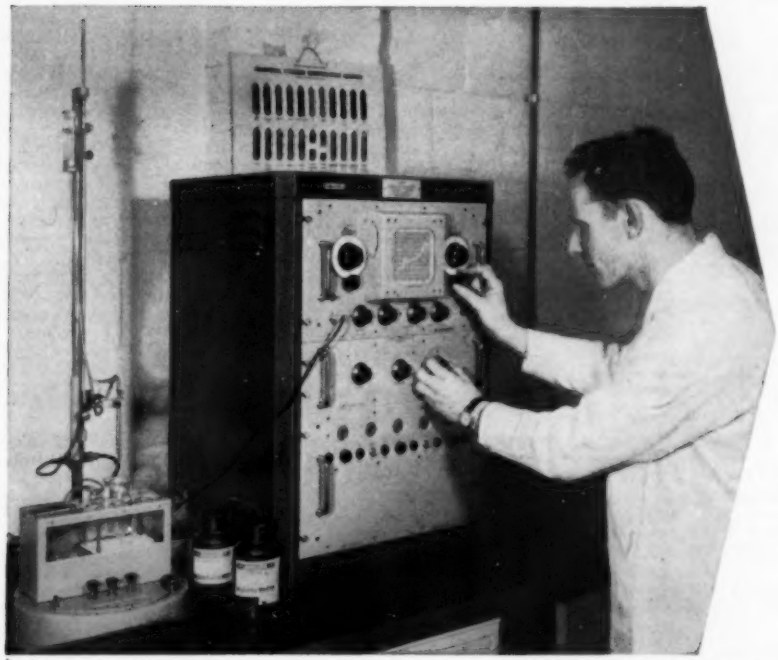
The Chemical Society (Cambridge). Joint meeting with the University Chemical Society. In the University Chemical Laboratory. 8.30 p.m. 'Biphenylene and Related Compounds', by Professor W. Baker, D.Sc., F.R.S.

The Chemical Society (Newcastle upon Tyne). In the Chemistry Department, King's College. 5.30 p.m. Bedson Club Lecture, 'The Gibberellins, a New Group of Plant Hormones', by Dr. P. W. Brian, F.R.S.

The Society for Analytical Chemistry (North of England Section). Joint meeting with the Manchester Section of the R.I.C. At the Manchester Literary and Philosophical Society, 36 George Street, Manchester 1. 2 p.m. 'The Design and Construction of Laboratories', by R. R. Young, F.R.I.B.A. and P. J. Harrington. 9 a.m. (from central Manchester) visit to I.C.I. (Pharmaceuticals Division), Alderley Park, Cheshire.

Incidental information

No. 16



Items of interest from our laboratory notebooks

Activated Alumina

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Unmasked end-point

***o*-Dianisidinetetra-acetic acid** is a fluorescent metal-indicator used for the complexometric titration of **metals that form coloured complexes.** Its brilliant blue fluorescence under u.v. is quenched by copper ions. These can therefore be used to back-titrate an excess of EDTA added to the ionic species to be determined. This indicator can also be used in the direct titration of **Ca** in alkaline solution. It is described by R. Belcher, D. I. Rees and W. I. Stephen, *Talanta* **4**, 78 (1960). Supplied from stock. CODE 3644·5. Price 1g 6/3, 5g 23/9.

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[1-(2-Hydroxy-5-methylphenylazo)-2-hydroxynaphthalene-4-sulphonic acid], another of our metallochromic indicators, is used for the EDTA titration of **Ca plus Mg.** Advantages are in the stability of its solution and its sharp colour-change. (*see* F. Lindstrom and H. Diehl *Anal. Chem.*, **32**, 1123 (1960)).
Available from stock. CODE 2795. Price 1g 6/-, 5g 22/9.

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The Society of Instrument Technology. (Fawley Section). In Room 4a, Administration Building, Esso Petroleum Company Ltd., Fawley. 5.45 p.m. 'Applications of Transistor and Diodes', by D. Osborne.

December 4

The Chemical Society (Cambridge). In the University Chemical Laboratory. 5 p.m. 'Some Hydrogen Transfer Reactions', by Professor H. B. Henbest, Ph.D, F.R.I.C.

The Society of Instrument Technology (South Yorkshire Section). At the University, Department of Fuel Technology, Mappin Street, Sheffield. 7 p.m. 'Problems of Flow Measurement', by H. E. Farrer.

December 5

The Chemical Society (Leicester). Joint meeting with the University Chemical Society. In the University. 4.30 p.m. 'Some Recent Observations on the Activity of Metal Catalysts', by Professor C. Kembell, M.A., Ph.D., F.R.I.C.

December 7

The Chemical Society (Aberystwyth). Joint meeting with the University College of Wales Chemical Society. In the Edward Davies Chemical Laboratories. 5 p.m. 'Microcalorimetry and the Thermogenesis of Living Species', by Dr. H. A. Skinner, B.A.

The Chemical Society (Manchester). In Room F1, Manchester College of Science and Technology. 6.30 p.m. 'The Structure of Proteins', by Dr. M. F. Perutz, F.R.S.

The Society of Instrument Technology (Tees-side Section). At the Cleveland Scientific and Technical Institution, Middlesbrough, 7.30 p.m. 'Electronic Controllers', by A. H. Isaac, B.Sc., A.M.I.Chem.E.

December 8

The Chemical Society (Aberdeen). Joint meeting with the Royal Institute of Chemistry and the Society of Chemical Industry. At Marischal College. 8 p.m. 'Inorganic Heterocycles', by Dr. N. L. Paddock, B.A.

The Chemical Society (Glasgow). Joint meeting with the Royal Institute of Chemistry, the Society of Chemical Industry and the Society for Analytical Chemistry. In the Royal College of Science and Technology. 4 p.m. 'The Structure of Natural Products by Direct X-ray Analysis', by Professor J. Monteath Robertson, D.Sc., F.R.I.C., F.R.S.

The Society for Analytical Chemistry. (Scottish Section). Joint meeting with the Chemical Society, the Glasgow Section of the S.C.I. and the Glasgow and West of Scotland Section of the R.I.C. At the Royal College of Science and Technology, George Street, Glasgow. 7.15 p.m. 'The Structure of Natural Products by Direct X-ray Analysis', by Professor J. Monteath Robertson, M.A., Ph.D., D.Sc., F.R.S.

The Society for Analytical Chemistry (Western Section). Joint meeting with the Cardiff and District Section of the R.I.C. At University College, Cardiff. 'Analytical Research', by J. Haslam, D.Sc., F.R.I.C.

The Society of Instrument Technology (Midland Section). In the Lecture Theatre of the Byng Kendrick Suite of the Gosta Green College of Technology, Aston Street, Birmingham. 6.30 p.m. for 7 p.m. 'Instruments in Clinical Chemistry', by N. Crawford. 'Electronics in Surgery', by R. Lightwood.

Personal Note

Dr. D. H. Sharp

Dr. D. H. Sharp has been appointed Director (Technical) of the F.B.I. as from November, 1, 1961. He was to take up the appointment on the retirement of the present Technical Director, Major-General A. J. H. Dove, who left the Federation at his own request at the end of October in order to devote more time to voluntary work.

As Director (Technical) of the F.B.I. he will be responsible for the administration of a wide range of F.B.I. policy and services, including the following: location of industry; town and country planning; fuel; transport; water supply; trade effluent disposal; industrial research; education; industrial art; technical aid to developing countries; the F.B.I. overseas scholarship scheme; technical legislation.

The Electronic Computer Exhibition

The Second Electronic Computer Exhibition and Electronic Data Processing Symposium were held at Olympia, London, October 3-12. The first exhibition took place in 1958, and in the intervening three years many developments have occurred, the theme of this year's show being the work of the new generation of computers.

Faster machines, using ferrite core stores, are among recent developments. There have been improvements in input and output equipment, which is much faster than it was three years ago. New storage devices improve the location of specific information, and modular equipment enables a system to be built up in stages. Time sharing machines make it possible to handle more than one job at a time, while replaceable printed circuitry reduces the need for maintenance and automatic self checking devices increase reliability.

Another interesting trend is towards centralized installations, and the Post Office is developing services to meet the demand. On the GPO stand various types of transmission facilities were demonstrated or explained, including private circuits and equipment for preparing, transmitting and receiving punched paper tape using the standard international 5-unit code, and the public Telex network.

While the main emphasis was on commercial applications there was much of specific interest to the scientist and research worker.

The latest version of the National Elliott '803', for example, was seen solving problems in the fields of scientific statistics and operational research. Mathematical programmes demonstrated on the IBM 1620 data processing system included solution of simultaneous equations and regression analysis. In addition to a scale model of the machine itself, the control desk of the first production model of the Ferranti Atlas computer was exhibited. This computer, now being installed at Manchester University, is a general purpose machine suitable for scientific calculations as well as data processing.

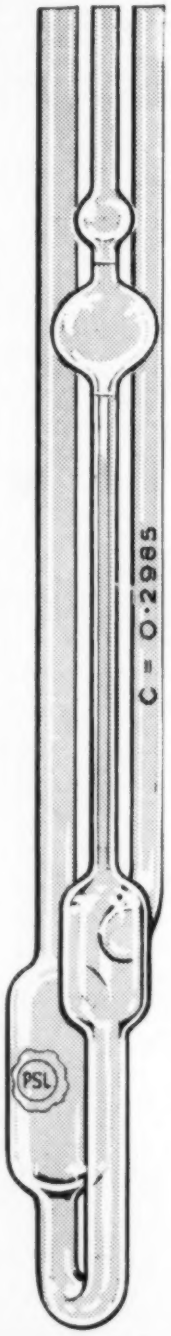
Analogue computers and analogue-to-digital converters were shown by a number of firms. Short Brothers & Harland exhibited an educational computer developed to meet the present trend towards the introduction of computing techniques into the curricula of Universities and technical colleges. The computer is used with a separate oscilloscope and power units as these are usually available in most technical colleges and electronic laboratories.

Despite the glittering array of equipment, at least two speakers referred to Britain's backwardness in the use of computers.

Lord Brabazon of Tara, who opened the exhibition, said that in electronics Britain had led the world. 'We were first with television, first with radar. But in the field of computers we have fallen behind America and the Continent of Europe, not in making the computers but in selling them. Or, to put it more truly, in applying them.'

Dr. Stanley Gill, in his paper on 'The Place of the Programmer', remarked that ten years ago Britain led the world in computers. Since then we had fallen sadly behind the U.S.A., 'due chiefly to a failure to appreciate the true magnitude and generality of the new technology.' Professional mathematicians had mostly been slow to seize the opportunities that computers offered.

It is to be hoped that the Electronic Computer Exhibition has succeeded in fostering a new attitude of mind that will help Britain to regain her original position in this new and fascinating field. M.M.



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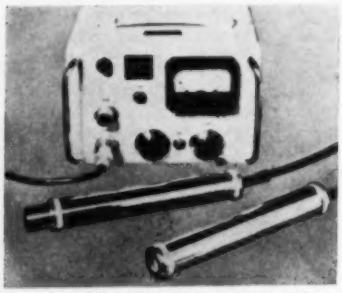
New Selective Gamma Monitor

A compact selective gamma monitor, Type PNI 1118, capable of detecting small quantities of those radioactive materials emitting gamma rays in the energy range 10 KeV. to 1·5 MeV. is now on the market. The monitor detects these rays by means of a scintillation counter followed by a single channel pulse amplitude analyzer. All circuits employ transistors. The monitor is mains or vehicle battery operated, is readily transportable, and is provided with 6 ft. of connecting cable between the indicator unit and detector probe.



This selective gamma monitor Type PNI 1118 has been introduced to detect small quantities of those radioactive materials emitting gamma rays in the energy range 10 KeV. to 1·5 MeV.

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Threshold bias is preset for the single channel pulse amplitude analyzer, and channel width is switched to give 50, 25 and 10 per cent channels on the 10-60 KeV, 50-300 KeV. and 0·25-1·5 MeV. ranges respectively. Channel widths are preset internally.

(Manufacturers: Plessey Nucleonics Ltd., Weedon Road, Northampton.)

Ion Exchange Progress

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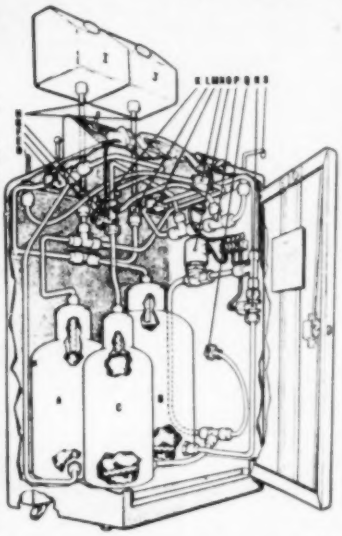

A, Prepurifier columns. C, Elgalite C.209 mixed bed cartridge. D, Mains water influent. F, Cation charging fluid inlet. G, Conductivity cell. I, Cation charging fluid tank. J, Anion charging fluid tank. L, Anion charging fluid inlet. N, Control valve. Q, Mixed bed cartridge drain tube. R, Prepurifier line cell. S. Effluent point.

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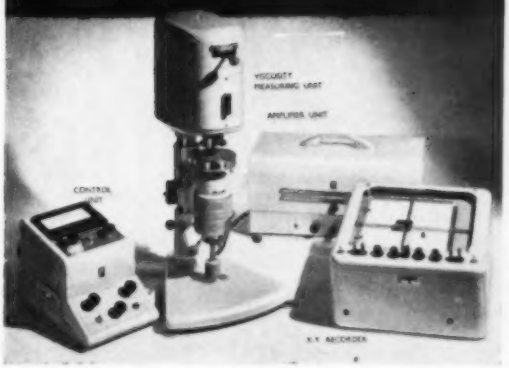

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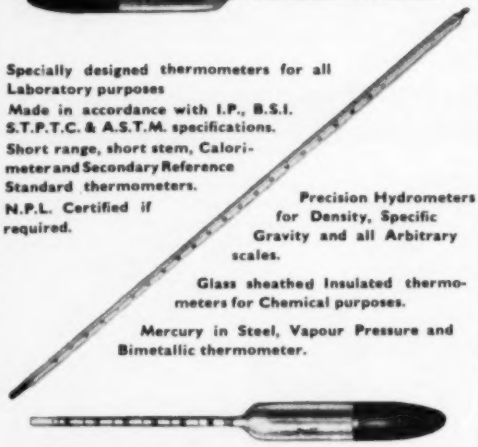

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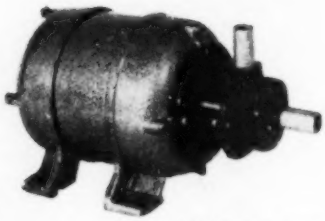

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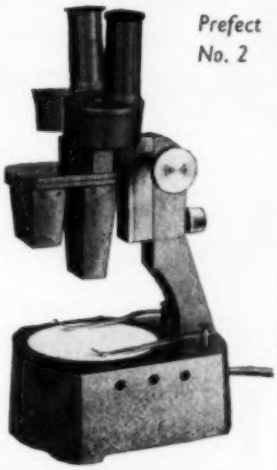

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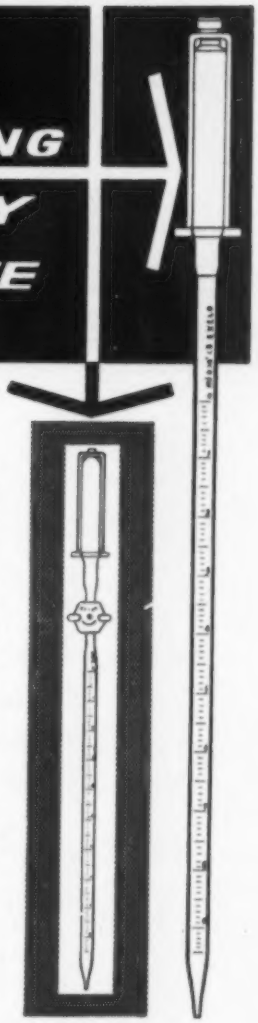

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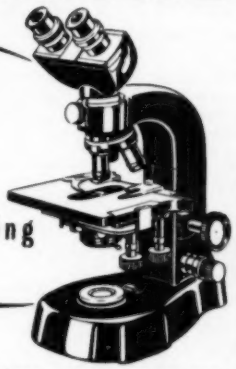

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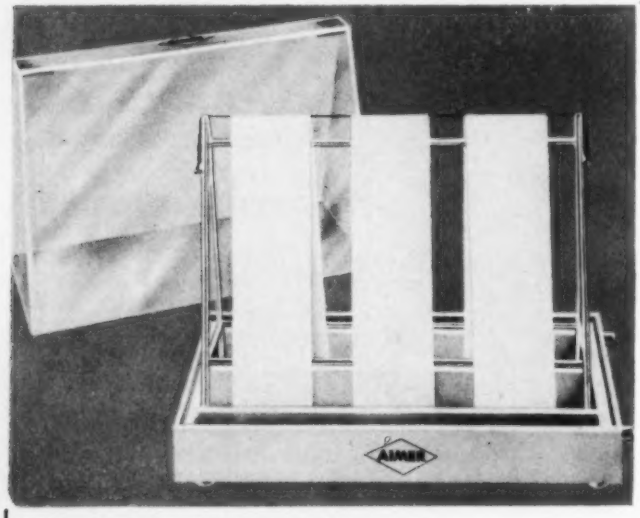

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Applications close, in New Zealand and London, on 15*th November*, 1961.

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Applications to be addressed to the Unit as above at the Department of Physiology, Medical School, Birmingham 15, quoting reference T.3.

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